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**JOURNAL OF SCIENCE**

*A Quarterly of Research*



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# ABSTRACTS OF DOCTORAL THESES<sup>1</sup>

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<sup>1</sup> Complete copies of these theses may be consulted at the Library, Iowa State College, Ames, Iowa.



# TRANSIENT RESPONSE OF FOUR-TERMINAL NETWORKS<sup>1</sup>

WILTON ROBERT ABBOTT

*From the Department of Electrical Engineering, Iowa State College*

This research is concerned with the treatment of four-terminal networks as a class, and the development of relationships which apply to all members of the class.

There are several methods of attacking transient problems. The method chosen here is that of the Laplace Transform. In this method a function of time,  $t$ , is transformed to a function of a complex variable,  $s$ , according to the following transformation.

$$(1) \quad F(s) = \int_0^{\infty} f(t) e^{-st} dt$$

The following theorems give methods of carrying out the transformation (1) for certain networks without having to go through the integration process.

The results of Theorem I are used in many places without benefit of formal proof.

**THEOREM I.** If the ratio of the a-c response to the excitation in a linear passive network is known as a function of  $j\omega$ , then the ratio of the response transform to the excitation transform is found by substituting  $s$  for  $j\omega$  whenever there is no initial energy storage in the network. The converse is also true.

Networks which are passive but include vacuum tube amplifiers operated class A are defined as "class A" networks. In considering such networks it is found that they differ from ordinary passive networks only by the presence of unsymmetrically placed off-diagonal terms in the conductance matrix. The unsymmetrical terms differ from the corresponding terms for the passive network by the transconductance of the vacuum tube. Mathematically the vacuum tube is equivalent to a negative, unilateral conductance. Considering these facts, Theorem II can be proven.

**THEOREM II.** If the ratio of the a-c response to the excitation in a class A network is known as a function of  $j\omega$ , then the ratio of the response transform to the excitation transform is found by substituting  $s$  for  $j\omega$  whenever there is no initial energy storage in the circuit. The converse is also true.

Theorems I and II make it possible to make use of any available information as to the a-c response of the network in evaluating its transient response. Such information for the passive network is given

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<sup>1</sup> Doctoral thesis No. 782, submitted December 15, 1945.



in some detail in Guillemin<sup>2</sup>. This work needs to be scrutinized carefully in the class A case as Guillemin often makes use of symmetries which exist in passive but not class A networks. The results give several methods of relating the output voltage and current to the input voltage and current in four-terminal networks. The most useful of these relationships is given in equation 2.

$$(2) \quad \begin{vmatrix} V_1 \\ I_1 \end{vmatrix} = \begin{vmatrix} A & B \\ C & D \end{vmatrix} \times \begin{vmatrix} V_2 \\ -I_2 \end{vmatrix}$$

In this equation  $V_1$  and  $I_1$  are the input voltage and current and  $V_2$  and  $I_2$  are the output voltage and current.

$$(3) \quad \begin{vmatrix} A & B \\ C & D \end{vmatrix} = -\frac{1}{y_{21}} \begin{vmatrix} y_{22} & 1 \\ y_{11} y_{22} - y_{12} y_{21} & y_{11} \end{vmatrix}$$

The admittance  $y_{21}$  is the short circuit transfer admittance at terminal pair 1;  $y_{11}$  is the short circuit input admittance at terminal pair 1;  $y_{12}$  is the short circuit transfer admittance at terminal pair 2, and  $y_{22}$  is the short circuit input admittance at terminal pair 2.

As an example of the application of the preceding material the transient response of a shunt peaked wide band amplifier with negative feedback was calculated. It is known<sup>3</sup> that with no feedback such an amplifier gives its best transient response when<sup>4</sup>  $Q = 0.36$ .

The theoretical analysis showed that with a feedback factor equal to  $G$  divided by the transconductance of the amplifier tube the output was reduced by a factor of two, the time of rise to the final value was reduced 45 per cent, and the overshoot past the final value was increased from 1 per cent to 3 per cent. This is a worth-while improvement.

Experimental results checked very closely with theoretical results for the amplifier without feedback. Because of insufficient information concerning the equipment used it was impossible to duplicate experimentally the arrangement postulated in the analysis of the amplifier with feedback. The discrepancy in the results was accounted for qualitatively.

The Superposition Theorem (Theorem III) makes it possible to use the preceding methods when treating class A networks containing more than one independent energy source.

**THEOREM III.** In a class A network containing several independent sources the response caused by all the sources acting simultaneously equals the sum of the responses caused by each of the sources acting separately.

<sup>2</sup>E. A. Guillemin. *Communication Networks*. Vol. 2. pp. 132-80. New York, John Wiley and Sons, Inc., 1935.

<sup>3</sup>H. E. Kallmann, R. E. Spencer, and C. P. Singer. Transient response. *Proc. Inst. Radio Engrs.* 33:169-95. 1945.

<sup>4</sup> $Q = LG/C$  where  $L$  is the peaking inductance,  $G$  is the load conductance, and  $C$  is the shunt capacitance of the amplifier.

Thevenin's Theorem (Theorem IV) is often very useful and should be considered in relation to any problem which may occur.

**THEOREM IV.** The current flowing in the load of a class A four-terminal network is the negative of the open circuit voltage divided by the sum of the load impedance and the reciprocal of the short circuit admittance seen from the output terminal pair.

# INTERPRETATION OF CHEMICAL AND BIOLOGICAL ANALYSIS OF VITAMIN C IN APPLES HELD UNDER VARIOUS CONDITIONS OF STORAGE<sup>1</sup>

ARDATH ANNA ANDERS

*From the Department of Foods and Nutrition, Iowa State College*

In the freak storm of November 11, 1940, temperatures throughout Iowa dropped from a maximum of 50 degrees to a low of zero in less than 24 hours. Ninety-five per cent of the trees in commercial apple orchards in 45 counties in central, western, and southern Iowa were killed and, as a result, the 1941 apple crop was less than one-tenth of the 1940 crop. In making recommendations for replanting orchards, horticulturists advised the use of hardy stocks. Nutritionists felt that the food value of the fruit of different varieties also should be considered. Of the nutrients present in apples, ascorbic acid probably is one of the most important. Even though apples are classed as low in vitamin C, they may become an important dietary source of the nutrient when consumed in large quantities. To date, no comprehensive report of the dietary value of the midwestern apple in respect to vitamin C has been published.

In the present investigation, an attempt has been made to evaluate the true nutritive value of the apple in terms of potential antiscorbutic potency when ingested by the human being.

Before any of the contemplated studies could be made, it was necessary to arrive at some decision as to what represented an adequate sample of apples for the estimation of mean ascorbic acid content. The great difference in the concentrations of vitamin C of individual apples taken from the same tree, and the even greater differences in the ascorbic acid content of apples produced by different trees, showed the importance of using a large number of apples in a sample and of having an equal distribution within the samples of apples derived from different trees. In addition, the apples from any one tree had to represent fruit picked from all sides of the tree because a marked difference existed in the concentration of ascorbic acid in apples from the north and the south sides of a tree. The average concentrations of ascorbic acid in successive samples each representing both the north half and the south half of each of ten trees were not statistically different from each other. A sample thus formulated provided a good base for the study of the effect of any processing treatment on the ascorbic acid content of this fruit.

The concentration of ascorbic acid in 29 different varieties of apples when mature and freshly picked was determined. The quantity of vitamin C present was estimated chemically with the use of 2, 6-dichlorophenolindophenol. The amount of dye decolorized when a measured quantity

<sup>1</sup> Doctoral thesis No. 788, submitted March 19, 1946.

of sample reacted with an excess of dye was determined photocolormetrically. The results obtained show that the concentration of ascorbic acid in all varieties of apples studied with the exception of the Willow Twig and its sport, the Red Willow, is fairly low, averaging about 6 mg. per cent. The Willow Twig, however, as analyzed over a period of three years, contained quantities of ascorbic acid ranging from 19 to 23 mg. per 100 gm. of tissue. The data seem to indicate that the concentration of ascorbic acid in apples is an inherent varietal characteristic. The importance of the Willow Twig apple as a source of vitamin C in the diet is apparent.

Because apples have good keeping qualities, large quantities are stored for use during the winter months when they become one of the important fruits in the average dietary. Whether or not apples which have been held in storage are a dependable source of vitamin C has been studied only in part heretofore. The relative stability of this vitamin in several varieties, therefore, was tested following different storage treatments. The data indicate that, in general, more of the ascorbic acid present in the freshly picked fruit is retained when the apples are held at 32°F. than when the storage temperature is higher. The stability of the ascorbic acid in apples during storage seems to be determined by the variety of apple, and in some cases by the storage treatment. It was observed that the vitamin C content of all varieties except the Willow Twig decreased rapidly during the first few weeks of storage. Thereafter the losses were small. In direct contrast to other varieties, a significant synthesis of ascorbic acid occurred in the Willow Twig during a storage period of seven months.

Whether or not analyses made in the chemical laboratory depict the actual nutritive value of a food has been questioned. The analytical method chosen in the present instance may not be specific for ascorbic acid if certain substances known as reductones are present. Reductones possess no nutritive value, but reduce the indophenol dye in the same manner as ascorbic acid. Furthermore, the method used measures the reduced ascorbic acid, but not dehydroascorbic acid, a substance also possessing antiscorbutic activity. For these reasons, the data obtained were evaluated in terms of dietary significance. It was found that small quantities of dehydroascorbic acid and traces of reductones may be present in freshly picked apples. In general, however, the error introduced by the reductones seems to be offset by the dehydroascorbic acid present, so that actually the determination with the indophenol dye gives a fair approximation of the true vitamin C value of the apple. It is very interesting that reductones could not be detected in the Willow Twig apples even after they had been stored for seven months. This finding is in line with the conclusion that the ascorbic acid in this variety of apple is remarkably stable. Determinations of the concentration of dehydroascorbic acid and reductones in other varieties after different intervals of storage are being made in the laboratory at the present time. It will be of considerable interest to learn if reduced concentrations of ascorbic acid

following storage are associated with the development of either dehydroascorbic acid or reductones.

The inherently high concentration of ascorbic acid in the Willow Twig indicates the possibility of producing through a breeding program varieties of apples as valuable as the tomato as contributors of vitamin C to the diet. The importance of such a program to enhance the nutritive value of apples is strengthened by the finding that 100 per cent of the ascorbic acid of the Willow Twig apple is absorbed by the human subject and is, therefore, available for metabolic utilization. Whether the ascorbic acid of the apple withstands the processes of mastication and digestion has been questioned, but ascorbic acid absorption curves based on the assimilation of pure ascorbic acid and an equivalent quantity of vitamin C in the form of apple tissue show that the chemical determination of the vitamin C concentration of this apple variety represents its true nutritive value.

# ECONOMIC PROBLEMS OF AN ADEQUATE DIET IN CANADA<sup>1</sup>

JAMES R. BOWRING

*From the Department of Economics and Sociology, Iowa State College*

Measurement at the national level of the nutritional adequacy of foods consumed by Canadians necessitates the adoption of certain standards. It is obvious that no set diet would be adequate for all people. Habits, tastes, and customs vary too much for that. However, a range between minimum<sup>2</sup> and optimum<sup>3</sup> with allowance for alternative choices of actual foods can go far towards providing the measurement which is necessary, if talk of food deficiencies is to make sense.

The dietary allowances adopted by the Food and Nutrition Board of the U. S. National Research Council in May, 1941, have been used as benchmarks for this analysis. In drawing conclusions from the outcome of such an analysis, it is necessary to appreciate the limitation imposed by a scarcity of scientific evidence of a number of human nutritional requisites, and the use of national averages without appropriate stratification by income, occupation, and related factors.

The results show that the average Canadian diet is deficient in those nutrients which can be most efficiently obtained from milk and milk products, green vegetables, fresh and citrus fruits. Additional evidence indicates deficiencies in the consumption of the protective foods group, which is closely related to family earnings and income distribution.

An increased consumption of milk, and, to a lesser extent, fruits and vegetables in Canada during the war years was aided by increased production under the incentive of controlled prices, production and consumption subsidies, and increased buying power. The intake of riboflavin and ascorbic acid in the Canadian diet, however, was still inadequate.

The supply of vegetables, fruits, and milk available for consumers is affected by certain common factors. These are the seasonal production pattern, variations in local productive capacity, the flow of imports, geographical limitations on distribution, fluctuating prices, the knowledge by consumers of relative food nutritive values, and the use of technological improvements in storage, canning, freezing, and dehydration.

The relative importance of agriculture's share in the Canadian national dividend is declining. This is indicated by the reduced proportion of agricultural goods exported of total exports, reductions in the proportion gainfully employed in agriculture, and a steady decline in the relative importance of the physical production occupation groups com-

<sup>1</sup> Doctoral thesis No. 787, submitted December 18, 1945.

<sup>2</sup> The minimum standard may be regarded as food intake sufficient to enable the recipient to lead a normal life without severe forms of deficiency diseases.

<sup>3</sup> The optimum is an ideal standard which cannot be improved upon and which represents a diet that provides the maximum health which foods make attainable.

pensated by increased importance of the service occupation groups. The continued absorption of surplus agricultural labor by further industrial expansion is necessary for the maintenance of per caput farm income.

One of the greatest disturbing influences both within the agricultural industry and in its relation to the national economy has been the variations in returns to farmers from year to year. The uncertainty associated with the variation has nutritional, sociological, and production effects.

There are similar variations and uncertainties in food supplies available for domestic consumption in both urban and rural areas. The strongest influence in this regard is the net income or net wage earnings available for food expenditures. Basically these two questions of agricultural producers' and consumers' welfare are part of the same problem. Given a national income and a distribution of that income which will enable consumers to purchase adequate food, then an increased demand will be reflected in increased incomes to producers. Similarly, stability in the demand for food will reduce fluctuations in returns to producers. As a large proportion of Canadian agricultural income is dependent on export markets, this can only be regarded, however, as a partial solution of the producer problem.

Pre-war studies in Canada indicate that the major influence on the purchase of foods is the scale of family earnings. Marked differences exist between the higher and lower income groups. Food consumption increased during the war years as the national income and the total number gainfully employed increased. The existence of this potential demand for food has implications both nutritionally and for its probable effects on producers' income.

Fluctuations in food consumption below a minimum standard can be prevented in major part by the maintenance of relatively full employment. Under the existing resource use, this will necessitate a continued high level of exports and the maintenance of a high domestic wage bill which will allow greater consumption expenditure by wage earners, particularly in the low income groups. In the event of business recession and conditions of unemployment, with decreases in real wages, the government should assume certain responsibilities for the maintenance of food consumption, by a food subsidy program related to the cost of an adequate diet and the income of the consumer. This will not only slow up the down-swing of business activity but will remove the nation's health from the influences of fluctuating industrial activity and export trade, and add some stability to agricultural producers' income.

Other causes of malnutrition in Canada which, while not directly related to income levels, are no less important, are custom and food habits, knowledge of relative food values, fluctuating supplies, and restricted imports. The institution of a government food subsidy program should thus be accompanied by an extensive educational program on nutrition and efficient food use. Seasonal variations in the supply of milk, milk products, vegetables, and fruits can be reduced and their consumption increased by greater use of the technological advances in

canning, frozen foods, dehydrated products, and storage. This will also solve many distribution difficulties. The introduction of improvements in this field can be hastened by publicity and the provision of cheap credit, or where necessary, direct government investment. Tariffs or other protectionist devices which restrict in any way the maximum use of United States sources of supply of citrus and other fruits should be removed. Special feeding of the nation's children should be a government responsibility.

The advantages gained during the war period of a centrally directed food program suggest the advisability of establishing a Canadian Ministry of Food. This ministry would protect consumers' welfare through its food consumption, would coordinate production goals and food requirements by outlook information and planning assistance, and direct food subsidy programs and educational measures deemed advantageous. Such a scheme would reduce nutritional deficiencies in Canadian diets, maintain food consumption, and while adding a certain stability to producers' income, would create an inflationary effect during the downward swings of future business cycles.



# A CYTOLOGICAL STUDY OF THE COSTAL MARROW OF THE ADULT HORSE AND COW<sup>1</sup>

M. LOIS CALHOUN

*From the Department of Veterinary Anatomy, Iowa State College*

In view of the increasing importance of bone marrow as a diagnostic agent, a study of the marrow of the adult cow and horse was undertaken to determine the normal cytological picture.

Fourteen head of cattle and seven horses served as experimental material for the study.

The ribs were chosen as the site for procuring the marrow in both species. The subject may be confined in stocks or restrained against one side of a stall. Little or no resistance is encountered ordinarily. The back and side of the animal should be brushed with a grooming brush and the operative territory wiped with a damp cloth to remove as much dust as possible. The general area was palpated until the rib having the least amount of fascia covering it was located. A sampling study suggested that the best results might be obtained by entering the most anterior rib not covered with muscle. Practical experience proved that more material could be obtained by entering the rib as high as possible and still avoiding the latissimus dorsi and serratus posticus muscles.

The chosen site was shaved or clipped closely, the area was washed with a soap solution, and iodine applied. A local anesthetic such as 2 per cent procaine hydrochloride was administered. When anesthesia was complete a short incision was made in the skin and then the fascia and periosteum was incised. A No. 487 Goodell-Pratt hand drill equipped with a straight shank 3/32" jobber's drill was used to bore into the marrow cavity. A point midway between the anterior and posterior borders of the rib should be chosen for insertion of the drill because there is danger of missing the marrow cavity completely if the drill goes through either border. Such an accident would entail the dangers of penetrating the thoracic cavity. The drill "gives" when it reaches the marrow cavity. The drill was removed from the rib and a cannula with stilet (needle trocar) with the same outside diameter as the drill was inserted into the drill hole. The stilet was removed and an air-tight 10 cc. syringe attached to the cannula. One cc. or less of marrow was drawn into the syringe. The marrow should be more viscous than blood and greyish-red in color.

The syringe was separated from the cannula and the marrow ejected into oxalate tubes. Dunham fermentation tubes had been prepared for the marrow since their small bore allowed the marrow and oxalate to be mixed more readily. One tenth of a cubic centimeter of a 2 per cent potassium oxalate solution per cubic centimeter of marrow was evaporat-

<sup>1</sup> Doctoral thesis No. 785, submitted December 18, 1945.

ed to dryness in preparing the oxalate tubes. Blood samples were obtained from the jugular vein prior to obtaining the marrow samples.

The samples were taken to the laboratory immediately and both blood and marrow smears made at once. Then total red and white counts were done on the blood and the amount of hemoglobin determined. No total counts were made on the marrow sample because the dilution with blood appeared to invalidate the results.

Osgood's modification of Wright's stain was used to stain the smears.

Three hundred cells were counted in the blood differentials and 500 enumerated for the marrow differential counts. A field was chosen in which there was an even distribution of nucleated cells, the cells did not overlap and staining was sharp. The edge of the smear was avoided and the cells were counted at random by going back and forth across the chosen area. The mitotic figures encountered per 500 cells also were recorded. The megakaryocytes were counted in a 300 square millimeter area. The myeloid-erythroid ratio was determined. Cytological studies were made of the marrow cells and color comparisons were made with colors in the Munsell Book of Color. Colored photomicrographs were made of the bone marrow smears to illustrate the various types of cells and colored drawings of the various cells were incorporated into a plate.

The myelogram for the cow (range and mean in per cent): stem cell: 0.0-5.0, 2.14; erythroblast: 11.8-42.8, 30.26; normoblast: 7.2-39.2, 21.69; total erythroid cells (E): 21.0-72.2, 52.66; promyelocyte: 0.0-6.8, 1.51; neutrophilic myelocyte: 10.4-32.0, 19.39; neutrophil: 1.2-12.2, 5.73; eosinophilic myelocyte: 1.8-10.4, 6.69; eosinophil: 0.0-7.6, 1.92; all basophils: 0.0-1.0, 0.34; total myeloid cells (M): 19.6-60.4, 35.59; monocyte: 0.0-7.6, 2.64; plasma cell: 0.2-2.0, 0.79; lymphocyte: 1.4-16.8, 6.68; megakaryocytes in 300 sq. mm.: 0-121, 25.14; mitoses per 500 cells: 9-11, 4.9; myeloid-erythroid ratio (M/E): 0.27-2.59, 0.676.

The myelogram for the horse (range and mean in per cent): stem cell: 0.4-3.4, 1.6; erythroblast: 8.0-32.0, 20.94; normoblast: 5.0-24.2, 13.71; total erythroid cells (E): 19.0-47.6, 34.66; promyelocyte: 0.0-5.0, 1.83; neutrophilic myelocyte: 26.2-56.0, 38.06; neutrophil: 1.8-20.2, 13.31; eosinophilic myelocyte: 0.4-3.6, 2.34; eosinophil: 0.2-1.2, 0.60; all basophils: 0.0-1.0, 0.60; total myeloid cells (M): 45.0-71.6, 56.74; monocyte: 1.2-4.8, 2.46; plasma cell: 0.0-0.8, 0.63; lymphocyte: 2.0-5.6, 3.91; megakaryocyte in 300 sq. mm.: 0-8, 1.71; mitoses per 500 cells: 0-8, 2.71; myeloid-erythroid ratio: 0.94-3.76, 1.64.

Graphs indicated a positive correlation between the marrow neutrophilic myelocyte and the adult neutrophil in the blood but no correlation between the marrow eosinophilic myelocyte and the eosinophil in the circulating blood. Similarly, graphs comparing the marrow red blood cell series to the erythrocytes in the peripheral blood suggested some correlation though not as striking as the neutrophil or eosinophil. Individual neutrophil curves for all the animals were similar. Figures were not available for the horse but the neutrophil curve of the cow agreed favorably with those of other investigators in the field.

A sampling study was made at two different levels on each of five ribs (8th-12th) in one cow and the cell counts recorded. An analysis of variance of the means showed a significant variation from the mean for the erythroblasts, promyelocytes, and lymphocytes in the 12th rib. A significant positive trend was observed from the eighth to twelfth rib in the total erythroid cells, and the neutrophils showed a negative trend in the same direction. Since only one animal was used, more work along the same line is needed to confirm these data.

A study was made of the healing process of the drill hole in horse ribs and photomicrographs made to illustrate the progress of repair. Repair of the bone was almost complete in 7 weeks and all external indications had disappeared long before that.

# NON-LACTOSE FERMENTING YEASTS AND YEAST-LIKE FUNGI FROM CREAM AND BUTTER<sup>1</sup>

STANLEY H. F. CHINN

*From the Department of Dairy Industry, Iowa State College*

In studying changes caused by yeast forms in dairy products, bacteriologists have given most of their attention to the lactose fermenting group, while the non-lactose fermenting group has been relegated to a more or less secondary position. This viewpoint is understandable since the former group usually is considered to be the cause of a number of undesirable changes, while the latter group is thought of as being rather inert and therefore of less importance. However, non-lactose fermenting yeasts and yeast-like fungi are known to occur in considerable numbers in certain samples of dairy products, and several types are known to be able to cause definite changes in dairy products under some circumstances. This study was undertaken as an attempt to isolate and characterize the non-lactose fermenting yeasts and yeast-like fungi from cream and butter and to classify them by the use of exhaustive technics which are now available.

In this study, 369 cultures of yeasts and yeast-like fungi were isolated from cream and butter. One hundred and thirty-nine of the cultures were obtained from 124 samples of cream, and 230 cultures were obtained from 203 samples of butter. Samples were taken at the four seasons of the year. Of the cultures isolated, 342 (92.7 per cent) were unable to ferment lactose and 27 (7.3 per cent) were able to ferment this sugar.

The non-lactose fermenting organisms were separated into 28 types on the bases of cell morphology and dimension, sporulation, chromogenesis, ability to form mycelium, types of mycelial formation, carbohydrate fermentation and utilization, nitrogen utilization, alcohol utilization, appearance of growth on agar slant, growth temperature, proteolysis on milk plates, and gelatin liquefaction. Of the 28 types recognized, 2 (3 cultures) were placed in the genus *Saccharomyces* as described by Stelling-Dekker (6), 2 (41 cultures) in the genus *Rhodotorula* according to Lodder (4), 9 (81 cultures) in the genus *Torulopsis* according to Lodder (4), 2 (9 cultures) tentatively in the genus *Pullularia* as defined by Berkhout (1), 1 (11 cultures) in the genus *Trichosporon* as proposed by Diddens and Lodder (2), and 12 (197 cultures) in the genus *Candida* as described by Langeron and Guerra (3), Diddens and Lodder (2), and MacKinnon and Artagaveytia-Allende (5).

The 14 types having the characteristics of recognized species or varieties were identified as *Saccharomyces cerevisiae* (Hansen) "Press-hefe a Delft," *Rhodotorula mucilaginosa* (Jørgensen), *R. mucilaginosa* var. *carbonei*, *Torulopsis molischiana*, *T. laurentii*, *T. rotundata*, *T. uvae*,

<sup>1</sup> Doctoral thesis No. 800, submitted June 10, 1946.

*Candida krusei*, *C. parakrusei*, *C. chalmersi*, *C. tropicalis*, *C. lipolytica*, *C. flarei*, and *C. zeylanoides*.

Six types could not be identified definitely with previously described species because of minor differences in characteristics or incomplete descriptions in the literature, but they may be regarded tentatively as being closely related to previously described species or as varieties of these species.

Eight types could not be identified with previously described species. On the basis of correlated differences in characteristics, new specific designations seem warranted. However, further studies, especially comparisons with known cultures such as those listed by Diddens and Lodder (2) but not yet available, should be made before describing them as new species.

Characteristics in addition to those found useful for taxonomic separation were studied. Lipolysis and action on litmus milk frequently vary within a type and are of importance to the dairy industry but apparently of limited taxonomic significance. No great significance can be attached to the colony characteristics, since irregularities occurred so frequently within some types. Heat resistance showed that not one of the cultures was able to survive 61.7° C. (143° F.) for 30 minutes.

The action of 47 strains of representative non-lactose fermenting yeasts and yeast-like fungi on cream at 21° C. was studied, using incubation for one week. Four types developed very pronounced defects such as rancidity, putridness, cheesiness, and uncleanness. Seven types developed fairly pronounced off-flavors such as moderate bitterness, moderate rancidity, and yeastiness. Six types caused only mild defects such as slightly yeasty, slightly rancid, slightly unclean, and slightly bitter flavors. Four types caused no defects. Slight variations of defect development occurred within some types. In a number of instances, where defects were developed, the presence of *Streptococcus lactis* enhanced the off-flavors.

The action of 47 strains of representative non-lactose fermenting yeasts and yeast-like fungi on unsalted butter was studied at 21° C. and at 4° C. After one week at 21° C., 2 types produced very pronounced defects such as rancid and cheesy flavors, 3 types developed fairly pronounced defects such as unclean and moderately cheesy flavors, 8 types caused mild off-flavors such as slight bitterness, astringency, slight acidity, slight uncleanness, and slight rancidity, and 8 types developed no defect. Slight variations of off-flavors occurred within some types. When the butter was held at 4° C. for one month, 1 type developed a very pronounced rancid defect, 10 types caused mild defects such as slightly unclean, slightly acid, slightly cheesy, slightly rancid, and astringent flavors, and 10 types did not cause any defect. Slight variations of defect development occurred within some types.

Non-lactose fermenting yeasts and yeast-like fungi are distributed widely in cream and butter and are capable of causing pronounced defects under some circumstances.

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# MEAT IN NUTRITION. XXVII. CONCENTRATION OF UREA NITROGEN, CALCIUM, AND PLASMA PROTEIN IN THE BLOOD OF PREGNANT RATS FED A DIET CONTAINING PARTIALLY DRIED, AUTOCLAVED PORK MUSCLE<sup>1</sup>

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A disorder of pregnancy in rats resembling the so-called toxemia of pregnancy in human beings has been produced by dietary manipulation in the Nutrition Laboratory at the Iowa State College. This disorder does not occur among gravid rats in the stock colony fed a ration composed largely of a mixture of cereal grains and milk, but makes its appearance regularly among pregnant females fed a semi-synthetic diet and containing partially dehydrated pork muscle as its chief source of protein, designated as Pork I.

One of the objectives of the present investigation has been the assembly of the characteristic symptoms and pathological findings of the gestational disturbance in rats, as reported by other investigators in the laboratory, to relate these observations to the syndrome in other species including the human being, to each other, and to interpret them in terms of recent developments in physiology.

The depression in total non-protein nitrogen constituents in the blood normally occurring in pregnancy is not characteristic of pregnant non-toxic pork-fed rats. Also, abnormally high concentrations of this fraction characterize the blood of rats fed the Pork I diet when pregnancy disease develops. An increased hydration of tissues is also associated with the experimental toxemia, abdominal and pleural cavities often containing considerable amounts of free fluid. The present investigation was undertaken to determine whether an increase in urea nitrogen was the fraction responsible for the increase in total non-protein nitrogen in the blood, and whether a lowered concentration of plasma proteins might be responsible for the increase in moisture content of the tissues. Prior to the present investigation, no work had been done on the concentration of inorganic constituents in the blood of the toxic animal. In the present study, the effect of feeding the pork-containing diet on the concentration of serum calcium and the concentration of this constituent in the serum of those animals developing toxic pregnancy was determined.

When young albino rats, Wistar strain A, had reached sexual maturity, each rat was assigned to one of four experimental groups and fed the specific diet chosen for that group. The groups were classified as follows:

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<sup>1</sup> Doctoral thesis No. 789, submitted March 19, 1946.

1. Virgin rats fed the regular stock colony ration, which served as the control diet,
2. Virgin rats given the experimental Pork I diet,
3. Pregnant females fed the stock ration, and
4. Pregnant rats fed the Pork I diet.

Blood was drawn from the abdominal aorta at the beginning of the twenty-second day of the second pregnancy in the case of gravid rats and from virgins when they were of approximately the same age as the pregnant animals. A portion of the blood was oxalated and used for the determination of the respective concentrations of urea nitrogen and plasma protein. The rest of the blood was allowed to clot and the concentration of calcium in the serum determined. At necropsy, the physical condition of the rats was noted and the organs examined in a systematic fashion.

Group comparisons were made of average values for the concentrations of urea nitrogen, plasma protein, and serum calcium. These comparisons showed two things, *i.e.*, the effect of pregnancy *per se* in both normal and experimental animals and the influence of the feeding of the pork diet to gravid and non-gravid rats.

The results of the study are summarized below:

- A. Pregnancy *per se* in the normal animal was associated with:
  1. A highly significant decrease in the concentration of urea nitrogen in the blood,
  2. A highly significant decrease in the concentration of protein in the plasma, and
  3. A highly significant decrease in the concentration of serum calcium;
- B. The feeding of the pork diet was associated with:
  1. A significant increase in the concentration of urea nitrogen in the blood of the pregnant animal over that characteristic of the control pregnant animal,
  2. No significant change in the concentration of urea nitrogen in the blood of the virgin animal,
  3. No significant change in the concentration of protein in plasma in either virgin or pregnant animals, and
  4. No significant change in the concentration of calcium in serum in either virgin or pregnant animals.

It is important to note that the only significant change in the concentration of the constituents studied in the non-toxic rats, which could be associated with the feeding of the Pork I diet, was a lack of depression in the concentration of urea nitrogen in the blood of the pregnant rats.

The concentrations of the same constituents was also determined in the blood of pregnant pork-fed rats that developed the toxic syndrome. In these animals, the concentration of urea nitrogen in the blood was abnormally high, the concentration of plasma protein was lower than that characteristic of the gravid control rats, and the concentration of serum calcium was higher than that in the blood of gravid non-toxic rat.



Thus, it can be concluded that the feeding of the diet containing pork does not cause abnormal changes in the concentration of plasma protein or serum calcium in the blood of albino rats, either in the virgin state or during uneventful pregnancy. Following the feeding of this particular experimental diet, however, there is clear evidence of a break in normal metabolic processes related to the utilization of nitrogen following the feeding of the pork diet. That this disturbance is a fundamental contributing factor to the appearance of the toxic syndrome seems assured because it appears in the non-toxic and toxic animal. The extreme elevation of urea nitrogen in the blood of the latter, in part, may represent a secondary as well as a primary disorder.

# PREPARATION OF AMINO ACIDS AND DERIVATIVES AND THEIR EFFECT ON THE GROWTH OF *LACTOBACILLUS* *ARABINOSUS*<sup>1</sup>

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The occurrence of *d*-amino acids in the hydrolyzates of antibiotics such as gramicidin, tyrocidin, and penicillin raises the question of the possible effect of *d*-amino acids and simple derivatives on bacterial growth. In the work reported, isomers and racemates of alanine, valine, leucine, and tyrosine, and isomers of derivatives of valine, leucine, and tyrosine were prepared and tested for their effect on the growth of *Lactobacillus arabinosus*.

The amino acids were tested in the presence of a synthetic medium.<sup>2</sup> The derivatives were tested by Carol Houck Bollenback, who used a natural medium and serial dilutions. Both media were complete in that they were capable of supporting normal growth of the organisms. Growth in the case of the synthetic medium was determined by titrating the acid produced in 72 hours. In the case of natural medium, growth was determined by visual inspection after 24 hours.

The nutritional requirements of the organisms with respect to alanine, valine, leucine, and tyrosine were determined. Leucine and valine were found to be essential amino acids, while tyrosine and alanine were accessory. These results checked those reported in the literature.<sup>2</sup>

The isomers and racemates of alanine, valine, and leucine were tested at levels of 20 mg./ml. *d*-Valine, *dl*-valine, *d*-leucine, and *dl*-leucine inhibited growth while *l*-valine, *l*-leucine, *d*-, *l*-, and *dl*-alanine were without appreciable effect. Because of their low solubility, *d*-, *l*-, and *dl*-tyrosine were tested at concentrations of 6 mg./ml. Even at this level, an undetermined amount precipitated out after autoclaving. The amount that remained in solution showed no effect on the growth of *L. arabinosus*.

A concentration of 20 mg./ml. of amino acid resulted in an increase in titratable acidity quite apart from that produced by the growth of the organisms. It was therefore necessary to correct the titers of tubes containing this amount. This increase in acidity can be accounted for by the reaction of the amino acid and the detrose contained in the medium.<sup>3</sup>

Tests run on from 2–20mg./ml. of *d*-valine and *d*-leucine showed that these isomers were effective at lower concentrations than 20 mg./ml. *d*-Leucine showed a gradual increase in inhibition with increase in concentration over the range tested. *d*-Valine, on the other hand, showed no effect below 4 mg./ml. Another difference in the effect of valine and

<sup>1</sup> Doctoral thesis No. 802, submitted June 11, 1946.

<sup>2</sup> Kuiken, Norman, Lyman, Hale, and Blotter, Jour. Biol. Chem. 151, 615 (1943).

<sup>3</sup> Frankel and Katchalsky, Biochem. Jour. 35, 1034 (1941).

leucine was noticed in the results obtained with the racemates. *dl*-Leucine had an activity equal approximately to the *d*-leucine it contained. *dl*-Valine, however, had an activity approximately the same as that of an equal amount of *d*-valine.

If sub-inhibitory amounts of *d*-leucine were autoclaved together with a leucine-free medium, it was found that the resultant medium would support more growth than that of a medium in which the leucine was autoclaved separately and added aseptically. This effect was noticed in concentrations of 0.4–4.0 mg./ml. With lower concentrations, growth was approximately the same as in the leucine-free control, regardless of whether the *d*-leucine was autoclaved together with or separately from the medium. A conceivable explanation for the availability of *d*-leucine after it is autoclaved with the medium is the possibility of some racemization occurring in the presence of glucose at the autoclaving temperature.

In connection with the results obtained with alanine, valine, and leucine, it was pointed out that *d*-valine and *d*-leucine have been isolated from gramicidin. Alanine isolated from this source was of the *l*-configuration. It was also pointed out that valine and leucine are essential amino acids for *L. arabinosus*, while alanine is accessory. This suggested that the *d*-amino acids might compete with their specific antipodes and thus interfere with the growth of the organism. The activity of the *dl*-amino acids, however, would make it seem more likely that the competition was not specific, but was between the *d*-amino acid and all *l*-amino acids.

The possible correlation between the results with *d*-leucine, *d*-valine, and *d*-alanine and the steric effect of the amino acid side chains was also discussed. *d*-Alanine, with its small methyl side chain, had no inhibitory activity. *d*-Valine and *d*-leucine, with isopropyl and isobutyl side chains, showed marked inhibition. This was related to the work of Bergmann and co-workers<sup>4</sup> who pointed out the steric effect of voluminous side chains of the substrates in their studies on the antipodal specificity of proteolytic enzymes. In connection with this, a series of straight-chain amino acids were tested for their effect on *L. arabinosus*. The series consisted of glycine, *dl*-alanine, *dl*- $\alpha$ -amino butyric acid, *dl*-norvaline, and *dl*-norleucine. All were tested at concentration molecularly equivalent to 20 mg./ml. of leucine. The results showed that *dl*-alanine had no effect; *dl*-norvaline and *dl*-norleucine were both inhibitory to about the same extent; and *dl*- $\alpha$ -aminobutyric acid was slightly inhibitory. Glycine also was inhibitory. The effect of glycine could be reversed by alanine or pyridoxine. That of *d*-valine and *d*-leucine could not. The glycine effect was similar to that reported by Snell and Guirard for *Streptococcus lactis*.<sup>5</sup>

Optically active derivatives of valine, leucine, and tyrosine were prepared for the purpose of enhancing the activity of the *d*-amino acids. The derivatives tested included the *d*- and *l*- forms of formylvaline, for-

<sup>4</sup> Bergmann, Zervas, Fruton, Schneider, and Schleich, Jour. Biol. Chem. 109, 325 (1935).

<sup>5</sup> Snell and Guirard, Proc. Nat. Acad. Sci. 29, 66 (1943).

mylleucine, glycylleucine, phthalylleucine, phthalylvaline, leucine methyl ester hydrochloride, valine methyl ester hydrochloride, prolylleucine, prolylvaline, tyrosine ethyl ester hydrochloride, N-benzoyltyrosine, N-benzoyltyrosine ethyl ester, N-benzoyltyrosylamide, 3-aminotyrosine, and 3-nitrotyrosine.

Compounds not previously reported included phthalyl-*d*-leucine (m.p. 118–119°), phthalyl-*d*-valine (m.p. 113–114°), phthalyl-*l*-valine (m.p. 114–115°), *l*-valine methyl ester hydrochloride (m.p. 168°), *d*-valine methyl ester hydrochloride (m.p. 168°), prolyl-*d*-leucine (m.p. 225°), prolyl-*l*-valine (m.p. 221°), prolyl-*d*-valine (m.p. 220°), 3-nitro-*d*-tyrosine (m.p. 216°), 3-amino-*d*-tyrosine dihydrochloride (m.p. 156–158°), and *d*-tyrosine ethyl ester (m.p. 115–117°). Specific rotations for most of these compounds were given.

None of the derivatives seemed to show antipodal specificity. 3-Amino-*l*-tyrosine, 3-amino-*d*-tyrosine, *d*- and *l*-leucine methyl ester hydrochloride, and the phthalyl derivatives of both *d*- and *l*-valine and leucine showed more activity than did *d*-leucine, although none of the compounds were active at a concentration lower than 2 mg./ml.

# INFLUENCE OF SOYBEANS ON THE FLAVOR OF MILK, CREAM, AND BUTTER, AND ON THE BODY AND TEXTURE OF BUTTER<sup>1</sup>

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In 1940-41 and 1941-42, two experiments were conducted at the Iowa Agricultural Experiment Station: (1) to determine the time necessary for cracked soybeans to produce a maximum change in the flavor and quality of milk and butterfat, (2) to study the variations in certain butterfat constants throughout a cow's lactation period, and (3) to determine the feasibility of using the iodine number as a measure in ascertaining when a feed with a high oil content has exerted its maximum effect.

## FIRST EXPERIMENT

Twenty Holstein cows of the Station herd were paired into two groups so that each pair was similar as to age, size, stage of lactation, and production. The cows were fed alfalfa hay and a grain mixture, which was made up of cracked corn, oats, wheat bran, bonemeal, salt, and either 11.1 per cent linseed meal (mixture I) or cracked soybeans (mixture II). Both groups were fed mixture I during the preliminary period (70 days). At the close of this period the animals in group 1 continued on mixture I; while those in group 2 were changed to mixture II. These rations were fed continuously for 74 days and then reversed. For the remainder of the trial (49 days) group 1 received mixture II (cracked soybeans) and group 2 was fed mixture I (linseed meal).

Individual and composite milk samples were taken in glass bottles and scored for flavor at regular intervals during the experiment. Usually, the milk was scored within six hours after being drawn from the cows. At periodic intervals a representative aliquot milk sample from each group of cows was separated. Iodine numbers of the milk fat were determined according to the Hanus method given by the A. O. A. C.

Certain cows in group 1, which received mixture I (linseed meal) during the first of the experimental periods, consistently produced milk with a rancid flavor. When the milk from these cows was excluded from the composite milk sample, the flavor was improved considerably. On the other hand, the cows in group 2, which were fed mixture II (cracked soybeans), consistently produced milk quite free from rancidity. There was little change in the quality of the milk after the cows were switched to mixture I (linseed meal). However, the incidence of rancid milk produced by the cows in group 1 diminished after they were fed cracked soybeans in the latter part of the experiment. The data indicated that factors other than feed were operating to cause the high

<sup>1</sup> Doctoral thesis No. 784, submitted December 17, 1945.

incidence of rancidity in the milk of the cows in group 1 and that the individuality of the cow probably played an important role.

The following points may be emphasized from the results of this experiment:

1. The selection of cows to be used in studying the effect of feed on milk flavor should be based not only on such factors as breed, age, stage of lactation, etc., but also on the flavors of the milk they produce when fed the basal ration.

2. When changes in fat composition are to be studied, a control group of cows should be carried continuously on each of the feeds being studied, for the duration of the experiment.

3. Non-significant correlation coefficients were found between milk yield and fat yield, and the occurrence of feedy, oxidized, and rancid flavors in the milk.

4. Highly significant negative correlations of  $-0.61$  and  $-0.65$  between milk and fat yields, respectively, and the occurrence of flat flavor, indicate that as the total milk and fat production increases the tendency for the production of milk having a flat flavor decreases.

5. Likewise, a significant negative correlation of  $-0.52$  between age and the occurrence of flat flavor indicates that as the age of the cow increases there is less tendency for the production of milk having a flat flavor.

6. A non-significant correlation coefficient was found between age and the occurrence of feedy flavor in the milk.

7. The correlation coefficient of  $0.462$ , which is about  $.004$  less than the significant point, indicates that the tendency to produce rancid milk may increase with the age of the cow.

8. The significant negative correlation coefficient,  $-0.53$ , found between age and the occurrence of oxidized flavor indicates that the incidence of oxidized flavor is greater in the milk of young cows than in that of older cows.

9. In general, the maximum effect of a feed on fat composition, as measured by the iodine number, may be expected in approximately 15 days.

10. The data obtained indicate that a direct relationship exists between mean external temperature and the iodine number of the butter-fat produced.

## SECOND EXPERIMENT

In view of the results of the work just reported, the plan of the second experiment (1941-42) was modified in two ways. Certain features of the iodine number trends obtained in 1940-41 indicated the advisability of carrying two control groups of cows, one for each feed being studied. It also indicated that the effect of feed on the flavor of milk might be masked by the milk of certain cows which was constantly of an undesirable flavor. For this reason, twelve Holstein and four Ayrshire cows were selected on the basis of their milk flavor from a large

group of cows that was fed mixture I (linseed meal) during the preliminary period, and then divided into four similar lots. By the method of randomization the lots were subdivided into four equal groups, each of which was fed a predetermined grain mixture. Alfalfa hay and the same grain mixtures were fed at the same rates as in the first experiment.

During the first experimental period (39 days) groups 1 and 2 were fed, respectively, mixtures I (linseed meal) and II (cracked soybeans). Then the rations were reversed and fed for a period of 74 days. At the end of this second period the rations were again reversed and for 56 days the cows were fed the rations previously fed in period I. Groups 3 and 4 (control groups) were fed, respectively, mixtures I and II, for the duration of the experiment.

Composite milk samples were taken in glass bottles about every five days from a single milking of each group of cows, then scored. Group aliquot samples of cream for one day were obtained at 4-day intervals, heated (143° F. for 30 minutes) to inactivate the lipases, and then scored. Twenty-four hours later both the milk and cream samples were scored again. The iodine and thiocyanogen numbers of the butterfat from the cream also were determined. For these determinations it seemed advisable to utilize composite samples of one day's milk yield to determine changes in fat composition rather than samples from individual milkings. In order to measure possible changes in the quality of the cream and milk fat, pH determinations of the cream and butter serum were made and acid numbers of the butterfat were determined.

The following conclusions are drawn from the second experiment:

1. Highly significant correlations were found between the barn temperatures (recorded the same day, one day before, and two days before the samples were taken) and the iodine and thiocyanogen numbers of the butterfat.

2. The barn temperature recorded one day before the samples were taken had a closer correlation to changes in fat composition than that recorded the same day or two days before.

3. The maximum effect of feed on the changes in butterfat composition, as measured by the iodine and thiocyanogen numbers, appeared to be reached in approximately 20 days after the first change of feeds (in the initial stages of lactation), whereas the time necessary for the iodine and thiocyanogen numbers to measure the full effect of the succeeding changes of feed was not indicated by the data obtained.

4. Cows appear to adjust themselves to rations containing either linseed meal or cracked soybeans as 11.1 per cent of the concentrate mixture when fed over a long feeding period, so that the iodine numbers of their butterfat are of about the same magnitude with their differences remaining fairly constant. On the other hand, when these feeds are fed for a short period and then reversed it seems to disturb the fat metabolism of the cows, causing the differences between the iodine numbers of the butterfat to fluctuate rather widely.

5. The changes in iodine numbers seemed to be largely dependent on the changes in the oleic acid content of the butterfat.

6. Non-significant differences were found between the iodine numbers of the butterfat from cows alternated between the linseed meal and the soybean rations.

7. A significant difference was found between the iodine numbers of the butterfat from cows fed linseed meal or cracked soybeans continuously throughout the experiment at the rate of 11.1 per cent of the concentrate mixture. The iodine numbers of the butterfat from cows fed the linseed meal were generally higher than those of the butterfat from cows fed cracked soybeans.

#### GENERAL CONCLUSIONS

The results of the first experiment indicated that the maximum effect of a feed on the changes in butterfat composition, as measured by the iodine number, may be expected in approximately 15 days after a change of feeds. However, data obtained in the second experiment indicated that the full effect may be reached in approximately 20 days after the first change of feed (in the initial stages of lactation), whereas the time required for it to produce its full effect following succeeding changes of feed was not indicated. A comparison of the data obtained in corresponding experimental periods of both experiments partially explains these differences.

The data obtained in the first experimental period (January 7 to March 21) of experiment I corresponded fairly well with those obtained in the same period (December 15 to January 23) of experiment II with respect to the time necessary for the feeds to produce their maximum effect on the changes in butterfat composition. It is noted that the experimental period of the first experiment was much longer than the one of the second experiment. The second experimental period (March 21 to May 5) of experiment I and the corresponding one (January 23 to April 7) of experiment II were similar in that the iodine numbers of the butterfat from the cows did not maintain the fairly constant differences after the second feed change which were characteristic of them after the first feed change. Although it did seem that the differences between the iodine numbers of the butterfat from the cows fed mixtures I and II in the first experiment became fairly constant in approximately 15 days after the change of feeds, this consistency was maintained for only a short time after the second feed change. In this case, the second experimental period of experiment I was much shorter than that of the second experiment and as regards season and lactation period corresponded more nearly to the third period of experiment II. One common characteristic of the iodine numbers of both experiments during April and May is that they were more erratic with less constant differences than were obtained during the first experimental period.

The relationship between air temperature changes and the iodine number of butterfat from cows used in both experiments was studied.



The external temperature was used in the first experiment, while barn temperature was used in the second one. Results obtained in the first and second experiments indicate that air temperature changes affect the iodine number of butterfat. In both experiments a closer correlation was found between the temperature recorded one day before the samples were taken and the iodine number of the butterfat than that recorded the same day or two days before. Also, the temperature recorded the same day had a closer relationship to the iodine number of the butterfat than that recorded two days before.

# THE COMPOSITION OF THE WATER-SOLUBLE FRACTION OF SOME CERAMIC CLAY DEPOSITS OF IOWA<sup>1</sup>

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The water-soluble materials in ceramic clays and shales have been of interest to the manufacturers and users of structural clay products for many years. These soluble materials cause various difficulties in the manufacture of the ware and are responsible for the formation of the white scum or efflorescence often seen on brick and tile structures.

There are many ways by which soluble materials may be introduced into the various types of structural clay products. (a) Some materials originate from the products of the various reactions responsible for the transformation of igneous rocks into clays and shales and are present in the natural clay. (b) Others are introduced during the manufacture of the ware from the fuel used in the kiln, or from water used in processing the clay. (c) Others may be traced to the cement, sand, or water used in preparing the binder. (d) Still others may be introduced into the structure from ground water or by reactions of certain constituents in the ware with atmospheric gases.

The chemical composition of the water-soluble fraction of clays and shales and the efflorescences formed on structural clay products has been found to vary over wide limits. The alkali and alkaline earth metals are the most common among the metal constituents, while small amounts of iron and aluminum are usually present. Sulfur in the form of sulfate is the most common among the nonmetals, although nitrates, chlorides, borates, vanadates, and silicates have also been reported. It has almost universally been accepted that sulfur in the form of alkaline earth sulfates is the most common constituent. This has been found to be particularly true of the Iowa clays and shales.

The sulfur usually occurs in the natural clay in the form of pyrite or one of the other sulfides of iron. These have been partially or completely oxidized to sulfate during the natural weathering processes.

Among the various efflorescent minerals found in burned clay products is Epsom salt or epsomite ( $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ ). Magnesium, therefore, is considered as one of the chief sulfate "carriers" and it has been found<sup>2</sup> that a magnesium content as low as 0.1 per cent is sufficient to cause scumming.

In order to predict whether a clay will produce a ware which is liable to effloresce, a number of tests may be made, among which is the determination of the per cent of soluble salts. Several writers have de-

<sup>1</sup> Doctoral thesis No. 781, submitted December 15, 1945.

<sup>2</sup> Ries. *Clays, Their Occurrence, Properties, and Uses*. 3rd ed., p. 161. John Wiley and Sons, New York. 1927.

scribed methods of determining this. The majority of these methods consist of treating a weighed sample of the clay with a definite volume of solvent, filtering, and evaporating the solution to dryness. The weight of the residue is then used in calculating the per cent of soluble salts in the clay.

There has been little agreement among the various writers on the soluble salt determination concerning certain details of the procedure which are known to affect the weight of soluble material extracted from a clay. The following factors are known to influence the weight of residue obtained by extraction methods: (a) the condition of the sample, e.g., whether dried at room temperature or at elevated temperature; (b) the type of solvent used for the extraction, e.g., distilled water or a solution of an electrolyte; (c) the ratio of the weight of the sample to the volume of solvent used in the extraction; (d) the time and temperature of digestion; (e) the method of filtration; and (f) the temperature and time of drying the residue.

During the examination of a series of ten Iowa ceramic clays and shales, data were obtained showing the variation in weights of residues obtained by varying the clay-water ratio during digestion. When the weight of sample was plotted against the per cent of soluble salts (the volume of water remaining constant) it was found that the values for per cent of soluble salts decreased rapidly as the sample was increased to about two grams and then decreased more gradually, approaching a minimum. The clay-water ratios used in these experiments were 1:500 to 1:37.5. By comparing the curves obtained from the different clays, it is evident that no single clay-water ratio which will be suitable for all clays can be adopted for this determination.

In order to determine the composition of the soluble salt fraction of the clays, a 125-gram portion of each clay was boiled with 6 liters of distilled water for 30 minutes in pyrex flasks. The suspension was allowed to stand for 72 hours, then one liter of the suspension was siphoned off into each of 5 liter flasks, treated with macerated filter paper and filtered through a mat of paper pulp in a Buechner funnel. The clear solutions were evaporated to dryness in weighed beakers and then dried at 105°–110° C. until the weights had become constant or the loss in weight between two successive heatings had become insignificant. The residues were found to lose weight gradually over a long period of time. In some cases heating for 65 days was necessary before the final weight was taken.

The gradual loss in weight of the residues was found to be due principally to the gradual dehydration of Epsom salt and gypsum. Samples of these minerals were heated for 23 days at 105°–110° C. before their weights had become constant. At the end of this period, the residues were analyzed. The results of these analyses show that under the conditions of the experiment, Epsom salt ( $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ ) is converted into kieserite ( $\text{MgSO}_4 \cdot \text{H}_2\text{O}$ ) while gypsum ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ) is almost completely dehydrated.

Analyses of the original clays were made and also the residues ob-

tained from three successive extractions of each clay. The results of these analyses show that there is little or no correlation between the chemical composition of a clay and the composition of the residue obtained by a single extraction.

TABLE 1.

COMPOSITION OF ORIGINAL CLAYS AND THE SOLUBLE SALTS OBTAINED BY ONE EXTRACTION USING A CLAY-WATER RATIO OF 1:40

Sample	SiO <sub>2</sub>	R <sub>2</sub> O <sub>3</sub>	CaO	MgO	SO <sub>3</sub>
	Pct.	Pct.	Pct.	Pct.	Pct.
A (clay).....	49.91	19.34	7.01	4.35	1.27
A (residue).....	9.11	0.60	29.78	5.45	39.54
B (clay).....	63.28	23.78	1.20	1.41	0.06
B (residue).....	21.96	1.12	26.04	2.69	4.99
C (clay).....	61.33	25.67	0.44	1.66	0.04
C (residue).....	15.74	1.66	22.97	3.95	11.26
D (clay).....	58.33	28.06	0.82	0.66	0.12
D (residue).....	16.97	1.84	27.88	3.44	8.52
E (clay).....	58.22	26.98	0.70	1.42	0.32
E (residue).....	22.70	1.96	20.87	2.61	6.13
F (clay).....	57.42	27.53	0.43	0.69	2.31
F (residue).....	11.89	0.97	28.26	6.28	35.65
G (clay).....	62.91	25.58	0.49	0.58	0.45
G (residue).....	21.20	5.96	21.99	2.82	41.29
H (clay).....	44.11	34.64	0.38	0.71	8.76
H (residue).....	6.28	16.96	12.78	3.09	35.17
I (clay).....	54.79	29.02	0.84	2.30	0.01
I (residue).....	24.38	1.30	10.95	2.56	7.54
J (clay).....	31.24	12.71	16.61	9.18	0.03
J (residue).....	14.99	0.46	32.49	6.22	10.71

A method for the calculation of the per cent of soluble salts in clays was developed which is based upon the soluble sulfate content. Because sulfur has been found to be the principal constituent in clays which causes efflorescence, it was thought that the per cent of soluble salts obtained in this determination should represent the entire soluble sulfate content plus the various other constituents which are invariably present.

This method involves first, a determination of the total soluble sulfate content of the clay. A series of determinations is then made of the weights of total residues obtained by extracting the sample by different clay-water ratios. The residues from these extractions are then analyzed for the per cent of sulfate sulfur.

It was found by Shell and Cortelyou<sup>3</sup> that within certain ranges of concentrations of clay-water suspensions, the amount of soluble sulfate extracted is proportional to the clay-water ratio. If, then, a sufficient number of values are obtained for the weights or residues from different clay-water ratios, the weight of residue containing the total amount of soluble sulfate may be calculated. The following sample calculation, applied to a clay containing 0.24 per cent total soluble sulfate (as  $\text{SO}_3$ ) illustrates the method.

Total weight of soluble sulfate from a 125-gram sample:  $(125) (0.0024) = 0.3000$  gm. The weight of soluble sulfate (as  $\text{SO}_3$ ) extracted by a clay-water ratio of 1:40 was found to be 0.2926 gm., while the total soluble salts extracted by this clay-water ratio was found to be 0.7398 gm. The clay-water ratio necessary to extract 0.3000 gm. of soluble sulfate is calculated

as  $(40) \frac{(0.3000)}{(.2926)} = 41.0$  or 1:41. The weight of soluble salts extracted by

a clay-water ratio of 1:41 will be  $(0.7398) \frac{(41)}{(40)}$  or 0.7583 gm. which is the

weight of soluble salts forming the residue containing the total weight of soluble sulfate in the sample.

As most clays contain sulfur in the form of pyrite, precautions must be taken to prevent oxidation to sulfate during the extraction.

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<sup>3</sup> Shell and Cortelyou. Jour. Amer. Ceram. Soc. 26, 180 (1943).

# SMALL SCALE PRODUCTION AND SOME REACTIONS OF 2-METHYLFURAN<sup>1</sup>

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Previous work in these laboratories has shown that 2-methylfuran can be produced by vapor phase hydrogenation of furfural over a copper chromite catalyst at atmospheric pressure (1). Material recoveries were not good. The losses presumably were due to carbonization or adsorption on the charcoal carrier employed, since the apparatus used was shown to be gas tight. It was the purpose of the present study to improve the method of making methylfuran, as well as to investigate some reactions of this substance.

A very active and stable copper chromite (2) could be made if, in the decomposition step of its preparation, the temperature was controlled within the range 300°–350° C. When this catalyst was distributed on activated charcoal in a 1:1 ratio and placed in a laboratory hydrogenation unit, 90–95 per cent yields of methylfuran were obtained in one passage of furfural through the catalyst. To aid in the preparation of the catalyst, a small kiln was constructed to permit easy control of the temperature in the range of 300°–350°.

In the laboratory a 91.2 per cent over-all yield of methylfuran was secured in a run in which 23 grams of methylfuran were produced per gram of chromite used. The apparatus employed was essentially that of Burnette (1). An 80.2 per cent yield was obtained in a small pilot plant in which 23 pounds of methylfuran were produced per pound of chromite used. The pilot plant design differed from the laboratory unit only in the respect that a dry ice trap was not used to remove methylfuran from the recirculated hydrogen.

The activity of the chromite was dependent to some extent on the precipitation and extraction methods used in its preparation. Optimum conditions for precipitation, ignition, and extraction were not determined even though these variables were recognized as influencing catalyst activity. Other factors which might influence catalyst activity, but which were not investigated, are: (1) nature of the salts from which the catalyst is made, (2) the concentration of reagents used, (3) the amounts of chemicals employed, and (4) the nature of the stabilizer used. It was found to be more difficult to obtain an active catalyst in large scale preparations of the chromite because of the difficulties in duplicating experimental conditions. The chromite quickly deactivated if a stabilizing metal such as calcium or barium was not included in its preparation.

A hydrogen to furfural ratio greater than 10:1 was necessary for complete conversion of the furfural to methylfuran with one pass through

<sup>1</sup> Doctoral thesis No. 798, submitted June 10, 1946.

the catalyst. During laboratory runs the catalyst temperature was not a critical factor in the range of 200°–250°. Since deactivation of the catalyst occurs at temperatures above 250°, the operating range of 200°–230° was preferred in order to avoid possible chance of exposing the catalyst to a high temperature.

The mass rates of flow employed in the pilot unit were approximately three times those in the laboratory apparatus. When furfural was admitted to the catalyst at these high flow rates, temperature surges of 75° were measured because of the high heat of hydrogenation. These temperature rises affected subsequent hydrogenation, even though they were only of momentary duration. The surges could be eliminated effectively by reducing the mass rate of flow to that employed in the laboratory.

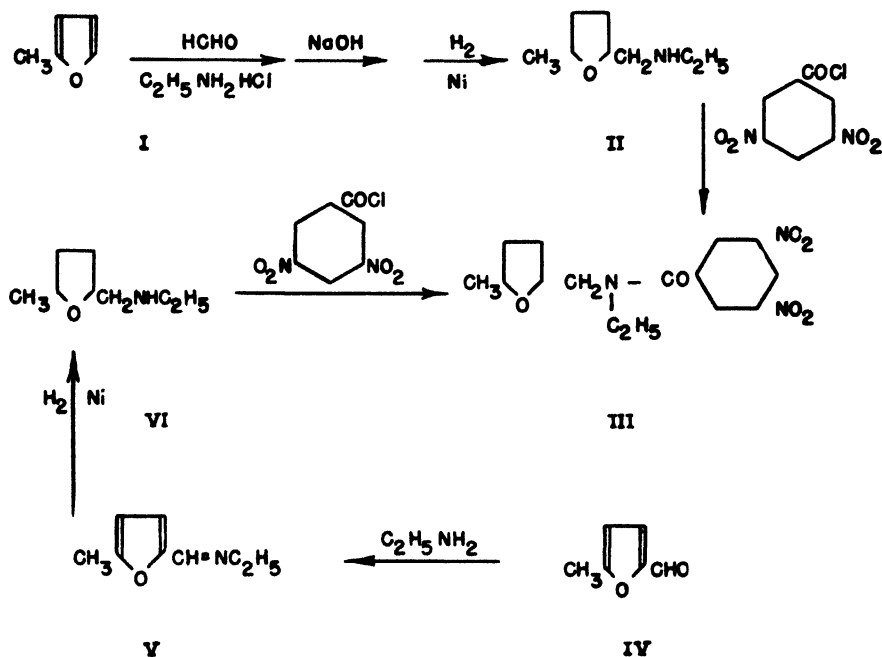
Data gathered in the laboratory indicated that if the methylfuran was not removed from the recirculated hydrogen it was absorbed to some extent on the catalyst. It seemed possible that this vapor would have to be removed from the recirculated hydrogen to prevent inactivation of the chromite in large scale operation. Vapor pressures at 20°, 25.6°, and 30° and vapor-liquid data at 738 mm. were determined on the furfural-methylfuran system in order that the required absorption and distillation equipment could be installed if necessary. A modified form of the apparatus of Smith and Menzies (3) was used to determine vapor pressures because it gave reasonably accurate results with a minimum expenditure of time. The apparatus used to determine vapor-liquid equilibria was essentially that of Othmer (4).

Methylfuran underwent the Mannich reaction. The fact that 2,5-dimethylfuran did not condense under similar conditions indicated that the reaction took place at the free alpha position in the methylfuran nucleus. Proof for the structure of the amine formed by the interaction of 2-methylfuran, formaldehyde, and ethylamine hydrochloride was obtained by the reaction sequence shown in the diagram.

5-Methylfurfural (IV) was condensed with ethylamine to give the imino derivative (V). Reduction of V with hydrogen and Raney nickel gave N-(5-methyltetrahydrofurfuryl)-ethylamine (VI) in 56 per cent over-all yield. The crystalline 3,5-dinitrobenzoate (III) of VI was identical with that prepared from II.

Thirteen primary and secondary amine hydrochlorides were condensed with methylfuran to give the corresponding Mannich bases. The reaction proceeded spontaneously with most of the amine hydrochlorides used. In the case of the primary amines, some tertiary amine was formed by further condensation of the initial reaction product with another molecule of formaldehyde and methylfuran. The tertiary amine was isolated and characterized in three instances.

Little attempt was made to find optimum conditions for the reaction. In one or two cases higher yields were obtained by cooling the reaction mixture to 30°–35° instead of allowing reflux temperatures to be reached. The acid-labile furan nucleus was polymerized by the hydrochlorides at the higher temperatures with a resulting decrease in yield.



Methylfuran has been polymerized in chloroform solution with concentrated mineral acids to give brittle, yellow-brown resins that aged badly in air. The resins could be stabilized by hydrogenation over Raney nickel but this treatment did not improve their physical properties. Methylfuran was found to react with formaldehyde, and ammonium chloride to give a nitrogen containing polymer. This resin may find possible applications as an ion-exchange resin.

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# PRODUCTION OF THE 2,3-BUTANEDIOLS BY THE FERMENTATION OF STARCH<sup>1</sup>

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2,3-Butanediol is a fermentation product which has received considerable attention in recent years. It can be converted to 1,3-butadiene, the basic material for the more important synthetic elastomers, and also offers many other possible uses in the synthesis of valuable organic compounds. The present thesis presents studies on the production of 2,3-butanediol by the action of *Aerobacter aerogenes* and *Aerobacillus polymyxa* upon appropriate substrates.

## METHODS

The organisms used in the investigations were: *Aerobacter aerogenes* B16, obtained from the American Type Culture Collection (ATCC No. 211); *Aerobacter aerogenes* B24, obtained from the culture collection of the Northern Regional Research Laboratory (NRRL B199); *Aerobacillus polymyxa* B25 (NRRL B510); and *Aerobacillus polymyxa* B32 (NRRL B510-R18). The stock cultures of *Aerobacter aerogenes* were carried on agar slants, and the stock cultures of *Aerobacillus polymyxa* were carried on liquid media containing 5 per cent of corn and 0.5 per cent of calcium carbonate. The fermented media were analyzed for 2,3-butanediol, ethanol, acetylmethylcarbinol, reducing sugars, and residual carbohydrate. The yields were expressed in terms of per cent of theory on the basis of carbohydrate utilized, and also in terms of per cent by weight of total carbohydrate available.

## EXPERIMENTAL RESULTS

### A. Production of 2,3-butanediol from dextrose by fermentation with *Aerobacter aerogenes*:

The effect of the addition of calcium carbonate and the effects of aeration and of agitation with nitrogen were studied. The addition of calcium carbonate definitely increased the rate of utilization of the dextrose without noticeably decreasing the yield of 2,3-butanediol. Aeration of the medium further increased the rate of dextrose utilization, as also did agitation of the medium with nitrogen. In fermentations conducted on a large laboratory scale, 35 per cent of the weight of dextrose present in a 16 per cent dextrose medium was converted to 2,3-butanediol in 93 hours.

### B. Production of 2,3-butanediol from corn by fermentation with *Aerobacter aerogenes*:

The possibility of utilizing brewery techniques and equipment for the production of 2,3-butanediol was investigated. Corn wort, prepared

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<sup>1</sup> Doctoral thesis No. 790, submitted March 19, 1946.

by filtration of malt-saccharified corn mash, was fermented with *Aerobacter aerogenes*. Lowering the saccharification temperature to 58° C., addition of manganous sulfate, and addition of malt to the fermenting medium effected increases in the yield of 2,3-butanediol. Thirty-five per cent (by weight) of the maltose present in the filtered wort was converted to 2,3-butanediol in a fermentation period of 42 hours. The results, obtained on a semi-pilot plant scale, show a definite possibility for large-scale production of 2,3-butanediol by adaption of brewery equipment and techniques to the fermentation process.

C. Production of 2,3-butanediol from corn by fermentation with *Aerobacillus polymyxa*:

The fermentation of unsaccharified corn mashes with *Aerobacillus polymyxa* was studied. Long periods of fermentation led to an increase in the yield of acetylmethylcarbinol and a decrease in the yield of 2,3-butanediol, particularly when the mash concentration was low. The weight per cent yields of 2,3-butanediol obtained from pre-thinned mashes containing up to 40 grams of corn per 100 ml. were nearly as high as the yields obtained from more dilute mashes. Nearly 30 per cent of the weight of the starch present in a 10 per cent corn mash was converted to 2,3-butanediol in 4½ days.

D. Production of 2,3-butanediol from corn starch by fermentation with *Aerobacillus polymyxa*:

The effect of the addition of inorganic and complex organic materials to the medium and the effect of the physical conditions of culture were investigated. The addition of a complex organic source of nitrogen was essential for high yields of the 2,3-butanediol. By the addition of a small amount of potassium permanganate to a medium containing corn starch, calcium carbonate, and corn gluten, the yield of the diol obtained was as high as that obtained in the fermentation of a corn mash equivalent in starch content to the starch mash. Yields of 2,3-butanediol obtained were nearly 27 per cent by weight of starch added.

Extensive subculturing of the organism had little effect upon the yields of 2,3-butanediol. Higher yields of the diol were obtained from mashes with high surface-volume ratios than from mashes with low surface-volume ratios.

The addition of malt sprouts and brewers' yeast to the inoculum resulted in higher yields of 2,3-butanediol than those obtained when these substances were not added to the inoculum.

E. Recovery of 2,3-butanediol from fermentation mashes:

The recovery of 2,3-butanediol from fermented media by solvent extraction was more efficient than recovery by distillation. By extracting a concentrated beer with diethyl ether in a continuous counter-current extractor, recovery yields were about 90 per cent of the 2,3-butanediol present in the fermentation medium.

# REARRANGEMENT REACTIONS OF SOME AROMATIC SULFUR COMPOUNDS<sup>1</sup>

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A historical survey of many rearrangement reactions of aromatic sulfur compounds has been given.

A further investigation was made of the rearrangement amination reaction that had been previously observed.<sup>2, 3</sup>

*o*-Bromophenyl methyl sulfide was rearranged by sodamide in liquid ammonia in 45.5 per cent yield to *m*-aminophenyl methyl sulfide. *o*-Chlorophenyl methyl sulfide was rearranged with potassium amide in liquid ammonia in 35.4 per cent yield to *m*-aminophenyl methyl sulfide. Based on the starting material actually used in the reaction, the yields of *m*-aminophenyl methyl sulfide were 57.5 per cent and 53 per cent, respectively. *o*-Chlorophenyl methyl sulfide was prepared in 92.5 per cent yield by the methylation of *o*-chlorothiophenol. The physical constants were: b. p., 116.5°–117° at 15 mm.;  $n_D^{20}$ , 1.6067;  $d_4^{20}$ , 1.2377.

*o*-Bromophenyl methyl sulfone was rearranged in liquid ammonia with sodamide to give 15 per cent of the *m*-aminophenyl methyl sulfone, based on the starting material actually used in the reaction. *o*-Bromophenyl methyl sulfone was prepared by the hydrogen peroxide oxidation of *o*-bromophenyl methyl sulfide in acetic acid. The yield was 94 per cent and the sulfone melted at 108°–108.5°. *o*-Chlorophenyl methyl sulfone was prepared in 88.5 per cent yield from *o*-chlorophenyl methyl sulfide; it melted at 93.5°–94.2°.

2-Bromo-4-methylbenzenesulfonamide and 2-iodo-4-methylbenzenesulfonamide did not undergo amination with sodamide in liquid ammonia. Several new compounds were reported in this series of preparations. 2-Bromo-4-methylbenzenesulfonic acid was prepared in 77 per cent yield from the 2-amino compound by diazotization and treatment with a hydrobromic acid-cuprous bromide mixture. The *p*-toluidine salt of this acid melted at 216°–218°. 2-Bromo-4-methylbenzenesulfonyl chloride melted at 64°–65°; methyl 2-bromo-4-methylbenzenesulfonate melted at 62°–63° whereas the ethyl ester melted at 70°–71°. The 2-bromo-4-methylbenzenesulfonamide, melting at 153.5°–154.5°, was prepared in 70 per cent yield from the crude sulfonyl chloride. 2-Iodo-4-methylbenzenesulfonic acid was prepared in 94 per cent yield by diazotization of the 2-amino compound and treatment of the diazonium salt with potassium iodide. The *p*-toluidine salt of the 2-iodo acid melted at 227.5°–230°. 2-Iodo-4-methylbenzenesulfonyl chloride, melting at 66°–67°, was

<sup>1</sup> Doctoral thesis No. 780, submitted August 27, 1945.

<sup>2</sup> Gilman and Avakian, *Jour. Amer. Chem. Soc.*, 67, 349 (1945).

<sup>3</sup> Gilman and Nobis, *ibid.*, 67, 1479 (1945).

prepared in 83 per cent yield from the potassium 2-iodo-4-methylbenzenesulfonate. 2-Iodo-4-methylbenzenesulfonamide melted at 165°–166°. 2-Acetamino-4-methylbenzenesulfonic acid was prepared in 74.5 per cent yield by the acetylation of the 2-amino acid. The *p*-toluidine salt melted at 218°–219.5°.

The addition products of sulfinic acids to benzalacetophenone were found to be excellent derivatives for several sulfinic acids. The melting point of these derivatives are as follows: *o*-Phenyl-*o*-benzenesulfonyl-propiophenone, 153.5°–154.5°; *o*-phenyl-*o*-(*p*-toluenesulfonyl)-propiophenone, 169°–169.5°; *o*-phenyl-*o*-(2-chloro-5-nitrobenzenesulfonyl)-propiophenone, 135.6°–136.4° (dec.); *o*-phenyl-*o*-(2-thiophenoxy-4-nitrobenzenesulfonyl)-propiophenone, 134.5°–136° (dec.). Magnesium methylsulfinate was prepared by the treatment of methylmagnesium iodide with sulfur dioxide. It was identified as its derivative, *o*-phenyl-*o*-methanesulfonylpropiophenone, which melted at 149°.

In cleavage reaction studies with sodamide in liquid ammonia, it was observed that diphenyl sulfone, diphenyl sulfoxide, and phenyl methyl sulfone were not cleaved. Only 78.5 per cent of the dibenzothiophene-5-dioxide was recovered from an attempted cleavage with sodamide in liquid ammonia; however, no sulfinic acid, the expected cleavage product, could be isolated from the reaction mixture. Ninety per cent of the thianthrene-9, 10-tetroxide was cleaved by sodamide in liquid ammonia; however, again the sulfinic acid, which was an expected cleavage product, could not be isolated. Since no phenylenediamine was obtained from the cleavage it appears that both sulfonyl groups were not cleaved. Steric effects might have prevented derivatization of the 2-aminobenzenesulfonyl-2-benzenesulfinic acid, probably produced by cleavage of the thianthrene-9,10-tetroxide.

Both dibenzothiophene-5-dioxide and thianthrene-9,10-tetroxide were produced in excellent yields, 98.5 per cent and 97 per cent, respectively, by the hydrogen peroxide oxidation of the parent compounds.

4-Nitro-2-sulfinodiphenyl sulfide was observed to have a melting point of 117°–118°, not the 135° reported by Krishna<sup>4</sup>. The 2-nitrothianthrene that was prepared from 4-nitro-2-sulfinodiphenyl sulfide melted at 134°–135.5°, instead of 128° as Krishna<sup>4</sup> reported. 1-Iodothianthrene was prepared by treatment of 1-thianthrenylithium with powdered iodine. The yield was poor (4.6 per cent) as considerable difficulty was encountered in the isolation of the product. It melted at 187.5°–188.5°.

2- and 3-Fluorodibenzofuran were prepared by treatment of the corresponding diazonium compounds with fluoboric acid and subsequent thermal decomposition of the fluoborates. The 2-isomer melted at 88.5°–88.8°, and the 3-fluorodibenzofuran melted at 88.5°.

3-Aminodibenzofuran was obtained in 99 per cent yield by the catalytic reduction of the 3-nitro compound by hydrogen in the presence of Raney nickel. 2-Nitro-3-aminodibenzofuran was also reduced cataly-

<sup>4</sup> Krishna, Jour. Chem. Soc., 123, 156 (1923).

tically by hydrogen in the presence of Raney nickel. The yield was 93.5 per cent.

The following series of new compounds was prepared in connection with the attempted preparation of 8-mercaptoquinoline-4-carboxylic acid. 4-Chlorocarbonyl-8-quinolinesulfonyl chloride was prepared in 92.5 per cent yield from 8-sulfocinchoninic acid and phosphorus pentachloride; it melted at 120.5°–126°. From this compound quinoline-4-carboxamide-8-sulfonamide, melting at 273°–277° (dec.), was prepared. Also methyl 4-carbomethoxy-8-quinolinesulfonate (m.p., 144°–147°) was prepared from 4-chlorocarbonyl-8-quinolinesulfonyl chloride. 4-Chlorocarbonyl-8-quinolinesulfonyl chloride was reduced with stannous chloride in concentrated hydrochloric acid to give the stannic chloride complex of 8-mercaptoquinoline-4-carboxylic acid. This complex could not be decomposed by acid or base to give the free 8-mercaptocinchoninic acid. The complex was methylated to give 8-methylmercaptocinchoninic acid (m.p., 215°–215.5°).  $K_a$  for this acid was  $2.6 \times 10^{-4}$ . Methyl 8-methylmercaptoquinoline-4-carboxylate was prepared by the esterification of 8-methylmercaptocinchoninic acid. Oxidation of the stannic chloride complex of 8-mercaptocinchoninic acid with iodine gave crude bis-(4-carboxy-8-quinolyl) disulfide. Pure bis-(4-carbomethoxy-8-quinolyl) disulfide was prepared by esterification of the disulfide diacid. The ester melted at 190.5°–191.5°. Pure bis-(4-carboxy-8-quinolyl) disulfide was prepared by saponification of the diester. It melted at 289.5°–290.5°; analysis indicated that the disulfide diacid was a tetrahydrate.

Nitration of 6-methoxy-2-phenylquinoline in fuming nitric acid gave a dinitro compound (m.p., 214.5°–215°) in 35 per cent yield and a mononitro compound (m.p., 156°–156.5°) in 17 per cent yield. The positions of nitration have not been proven but it is believed that nitration occurred in the benzo ring of the quinoline nucleus.

Nitration of 6-methoxy-2-phenylquinoline-4-carboxylic acid with potassium nitrate in sulfuric acid gave 9.9 per cent of a dinitro compound (m.p., 273°–275° dec.) and 60 per cent of a mononitro compound (m.p., 250°–251° dec.). Decarboxylation of the dinitro compound gave the same dinitro product that was obtained by nitration of 6-methoxy-2-phenylquinoline.

6-Methoxy-2-(*m*-nitrophenyl)quinoline-4-carboxylic acid was found to decarboxylate in 3.5 per cent yield when heated with copper bronze at 285° for three hours.

2-Nitro-4-aminoanisole was prepared in 60 per cent yield by the nitration of *p*-anisidine.

Benzal-3-nitro-4-methoxyaniline (m.p., 93.5°–94°) was prepared in 82 per cent yield by the condensation of benzaldehyde with 2-nitro-4-aminoanisole. Treatment of benzal-3-nitro-4-methoxyaniline with pyruvic acid did not give a nitro-6-methoxy-2-phenylquinoline-4-carboxylic acid but gave rather  $\alpha$ -phenyl-N-(3-nitro-4-methoxyphenyl)  $\alpha'$ ,  $\beta'$ -diketopyrrolidine- $\beta'$ -(3-nitro-4-methoxyphenyl)anil. This compound melted at 231°–232°.

# HIGH-MOLECULAR WEIGHT COMPOUNDS OF NITROGEN AND SULFUR AS THERAPEUTIC AGENTS<sup>1</sup>

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The large amount of lipoidal tissue present in certain strains of bacteria, such as the tubercle bacillus, suggested that the introduction into potentially physiologically-active substances of groups which would be lipoidal-soluble might give rise to valuable medicinals. This thought initiated the study and preparation of high-molecular weight compounds of nitrogen and sulfur as therapeutic agents.

General reviews of the chemotherapy of malaria and tuberculosis have been made, in addition to a survey of the physiological properties and therapeutic uses of compounds containing high-molecular weight aliphatic groups.

Two molar equivalents of dibutylamine and one molar equivalent of alkyl bromide were heated at 140°–155° for ten hours to give the following dibutylalkylamines: dodecyl<sup>2</sup> (68 per cent), b.p. 158°–160° (3.0 mm.);  $n_D^{20}$  1.4471;  $d_{20}^{20}$  0.8073; tetradecyl (76 per cent), b.p. 178°–180° (3.0 mm.);  $n_D^{20}$  1.4480;  $d_{20}^{20}$  0.8046; hexadecyl (81 per cent), b.p. 203°–205° (4.0 mm.);  $n_D^{20}$  1.4500;  $d_{20}^{20}$  0.8150; octadecyl (79 per cent), b.p. 204°–207° (3.0 mm.);  $n_D^{20}$  1.4513;  $d_{20}^{20}$  0.8192, becoming slightly turbid on standing. Dibutylaminomethyl butyl ether was cleaved by dodecylmagnesium bromide to give dibutyltridecylamine (52 per cent), b.p. 120°–122° (0.1 mm.);  $n_D^{20}$  1.448;  $d_{20}^{20}$  0.809; methiodide, m.p. 52.0°–52.5°. The preparation of octadecylchloride (87 per cent)<sup>3a</sup> and trioctadecylamine (52 per cent)<sup>3b</sup> is also given.

Rearrangement has been shown to occur during the reaction between  $\alpha$ -bromonaphthalene and lithium didodecylamide.  $\beta$ -N,N-didodecylaminonaphthalene (42 per cent) being obtained. This rearrangement is similar to that observed in the reaction between  $\alpha$ -halogenonaphthalenes and lithium diethylamide to form  $\beta$ -diethylaminonaphthalene.<sup>4</sup>  $\beta$ -Naphthylamine (3 equivalents) and dodecyl bromide gave  $\beta$ -N-dodecylaminonaphthalene (83 per cent)<sup>5</sup>, m.p. 41°–43°. Condensation of this secondary amine with dodecyl bromide gave  $\beta$ -N,N-didodecylaminonaphthalene

<sup>1</sup> Doctoral thesis No. 797, submitted June 10, 1946.

<sup>2</sup> German Patent, 611, 283 (1935) [C.A., 29, 4022 (1935)].

<sup>3</sup> (a) McCorkle, M. R., Doctoral Dissertation, Iowa State College (1938); (b) Hoyt, F. W., Doctoral Dissertation, Iowa State College (1940).

<sup>4</sup> Gilman, Crounse, Massie, Benkeser, and Spatz, Jour. Amer. Chem. Soc. 67, 2106 (1945).

Table 1  
MELTING POINT OF AMINE DERIVATIVES

Derivative	Derivatizing Agent	C <sub>14</sub> derivative m.p. and yield	C <sub>16</sub> derivative m.p. and yield	Mixed m.p. (C <sub>14</sub> and C <sub>16</sub> )
Benzenesulfonamide	Benzenesulfonyl chloride	66°-67° (79%)	71°-72° (92%)	62°-64°
Dithiourea	Carbon disulfide	80°-81° (92%)	87°-88° (quan.) <sup>a</sup>	78°-79°
Oxalamide	Ethyl oxalate	118°-119° (79%)	119°-120° (87%)	115°-116°
Phenylthiourea	Phenyl isothiocyanate	77.5°-78° (86%)	82°-82.5° (94%)	75°-76.5°
Urea	Potassium cyanate	112°-113° (88%) <sup>1</sup>	106°-107° (92%)	104°-105°

<sup>a</sup> Arnold and co-workers, Ber., 74, 1372 (1941).

<sup>1</sup> Buck, Hjort, Ide, and deBeer, Jour. Amer. Chem. Soc., 60, 461 (1938).

(37 per cent), b.p. 255°–260° (0.5 mm.);  $n_D^{20}$  1.531;  $d_4^{20}$  0.911; hydrochloride, m.p. 94°–95°. A mixed melting point of the hydrochlorides from the two preparations of  $\beta$ -N,N-didodecylaminonaphthalene showed no depression. o-Chlorophenyl dodecyl ether and lithium diethylamide in diethyl ether solution did not react on refluxing for 24 hours.

Tetradecylamine and hexadecylamine were purified by conversion to their hydrochlorides, followed by neutralization and distillation. Several derivatives of these amines were prepared. (Table 1).

The benzenesulfonamides are recommended as derivatives for tetradecylamine and hexadecylamine.

6-Chloro-5-nitroquinoline and sodium methyl mercaptide in methyl cellosolve at room temperature gave 5-nitro-6-quinolyl methyl sulfide (94 per cent), m.p. 138.5°–139°; at reflux temperature a very low yield of impure product was obtained. 6-Chloro-5-nitroquinoline and sodium dodecyl mercaptide in methyl cellosolve at room temperature for two and three-quarters hours and then at 60°–65° for two hours gave 5-nitro-6-quinolyl dodecyl sulfide (80 per cent), m.p. 44°–45°; this was reduced with Raney nickel catalyst to 5-amino-6-quinolyl dodecyl sulfide (76 per cent), m.p. 59°–60°; acetamido derivative, m.p. 121°–122°; dihydrochloride, m.p. 156°–157°. This amine would not react with salicylaldehyde, acetylacetone or *l*-isopropylaminopentyl chloride hydrochloride.

8-Acetamido-6-chloro-5-nitroquinoline and sodium methyl mercaptide in refluxing methyl cellosolve gave 8-amino-5-nitro-6-quinolyl methyl sulfide<sup>c</sup> (88 percent), m.p. 243°–244°. 8-Acetamido-6-chloro-5-nitroquinoline and sodium dodecylmercaptide in refluxing methyl cellosolve yielded 8-amino-5-nitro-6-quinolyl dodecyl sulfide (93 per cent), m.p. 89.5°–90.5°; acetylation with acetic anhydride and acetic acid gave 8-acetamido-5-nitro-6-quinolyl dodecyl sulfide (94 per cent), m.p. 67°–68°. Reduction of this product with Raney nickel catalyst at the temperature of steam gave 8-acetamido-5-amino-6-quinolyl dodecyl sulfide (80 per cent), m.p. 77°–78°, but the reduction product analyzed low for sulfur. This reduction product was acetylated with acetic anhydride and acetic acid to 5,8-diacetamido-6-quinolyl dodecyl sulfide, m.p. 126°–127°.

The preparation of 2-hydroxy-6-methoxylepidine (87 per cent), 2-chloro-6-methoxylepidine (72 per cent) and 6-methoxylepidine hydrate (98 per cent) is given.<sup>o</sup> 2-Chloro-6-methoxylepidine and sodium dodecyl mercaptide in refluxing methyl cellosolve gave 6-methoxy-4-methyl-2-quinolyl dodecyl sulfide (70 per cent), m.p. 73°–74°. 4,7-Dichloroquinoline and sodium dodecyl mercaptide yielded 7-chloro-4-quinolyl dodecyl sulfide (87 per cent), m.p. 59°–60°.

1-Diethylamino-2,3-epoxypropane was cleaved with sodium dodecyl mercaptide in refluxing toluene to give 3-diethylamino-2-hydroxypropyl

<sup>o</sup> Butterworth and Hey, Jour. Chem. Soc., 388 (1940).

<sup>c</sup> Gilman and co-workers, Jour. Amer. Chem. Soc., 68 (July, 1946).

<sup>\*</sup> Private communication, Dr. K. N. Campbell, University of Notre Dame, South Bend, Ind.



dodecyl sulfide (54 per cent), b.p. 151°–152° (0.2 mm.);  $n_D^{20}$  1.4739;  $d_{20}^{20}$  0.9049. The corresponding sulfoxide and sulfone could not be prepared by oxidation with hydrogen peroxide in acetic acid. 3,4-Epoxybutene-1 was cleaved by sodium dodecyl mercaptide to give 2-hydroxy-3-butenyl

dodecyl sulfide (77 per cent), b.p. 135°–137° (0.2 mm.);  $n_D^{20}$  1.4802;  $d_{20}^{20}$  0.9068; this product did not form an addition product with gold trichloride or platinum chloride. 3,4-Epoxybutene-1 and sodium hexadecyl mercaptide yielded 2-hydroxy-3-butenyl hexadecyl sulfide (84 per cent), b.p. 163°–164° (0.15 mm.); m.p. 26.5°–27.5°. Styrene oxide and sodium dodecyl mercaptide gave 2-phenyl-2-hydroxyethyl dodecyl sulfide (61 per cent), b.p. 178°–180° (0.2 mm.);  $n_D^{20}$  1.5130;  $d_{20}^{20}$  0.9062; methiodide, an oil. Styrene oxide and sodium hexadecyl mercaptide yielded 2-phenyl-2-hydroxyethyl hexadecyl sulfide, (58 per cent), b.p. 220°–221° (0.3 mm.);  $n_D^{20}$  1.5070;  $d_{20}^{20}$  0.9431.

Phenacyl dodecyl sulfide (85 per cent), b.p. 174°–178° (0.32 mm.); m.p. 34°–35°, was obtained from phenacyl bromide and sodium dodecyl mercaptide. This compound was also prepared in 35 per cent yield by the oxidation of 2-phenyl-2-hydroxyethyl dodecyl sulfide, using aluminum isopropoxide and cyclohexanone in refluxing toluene. The identity of the two products was proved by a mixed melting point of the 2,4-dinitrophenylhydrazone, m.p. 80°–81°. This proof of structure showed that in basic solutions epoxides are cleaved by mercaptides to give secondary alcohols rather than primary alcohols.

A mixture of myristonitrile and tetradecyl mercaptan, saturated with anhydrous hydrogen chloride, yielded tetradecylimido tetradecyl sulfide hydrochloride (65 per cent), m.p. 72°–73°. Laurophe none and phosphorus pentachloride formed 1-phenyl-1-chlorododecene-1 (88 per cent), b.p. 138°–140° (0.3 mm.);  $n_D^{20}$  1.515;  $d_{20}^{20}$  0.9647. Dodecylmercuric bromide,<sup>10</sup> prepared in 71 per cent yield from dodecylmagnesium bromide and mercuric bromide, and sodium thiosalicylate gave *o*-dodecylmercuri-mercaptopbenzoic acid,<sup>11</sup> (15 per cent), m.p. 72°.

Picolinic acid (52 per cent), m.p. 135°–136°, from the oxidation of  $\alpha$ -picoline by aqueous potassium permanganate, was esterified with ethanol using anhydrous hydrogen chloride to give ethyl picolate (73 per cent), b.p. 135°–136° (18 mm.). Condensation of this ester with ethyl acetate using sodium ethoxide yielded methyl 2-pyridyl ketone<sup>12</sup> (75 per cent), b.p. 79°–80° (10 mm.). Reaction of this ketone with isatin in ethanolic potassium hydroxide solution gave 2-(2'-pyridyl)cinchoninic acid (81 per cent), m.p. 302°–303°. This acid was esterified with ethanol and sulfuric acid to give ethyl 2-(2'-pyridyl)cinchoninate (85 per cent), m.p. 71°–72°.

<sup>10</sup> Meals, R. N., Doctoral Dissertation, Iowa State College (1942).

<sup>11</sup> Rumpf, *Bul. Chim.*, 9, 661 (1942) [*C.A.*, 38, 2951 (1944)].

<sup>12</sup> Gilman, Tolman, and Massie, *Jour. Amer. Chem. Soc.*, 68, (July, 1946).

The preparation of 6,7-methylenedioxy-2-phenylcinchoninic acid by the following synthesis<sup>13</sup> gave low yields: piperonal  $\rightarrow$  2-nitropiperonal  $\rightarrow$  5,5',6,6'-dimethylenedioxyindigotin  $\rightarrow$  3,4-methylenedioxyisatin  $\rightarrow$  6,7-methylenedioxy-2-phenylcinchoninic acid. The following synthesis gave better results. 3,4-Methylenedioxynitrobenzene, obtained in 14 per cent yield as a side-product in the nitration of piperonal, was hydrogenated with Raney nickel catalyst to give 3,4-methylenedioxyaniline (90 per cent), b.p. 97° (0.2 mm.); condensation of this amine with benzaldehyde gave 3,4-methylenedioxy-N-benzylideneaniline (96 per cent), m.p. 49.5°–50.5°. This anil was condensed with pyruvic acid in refluxing methanol to give 6,7-methylenedioxy-2-phenylcinchoninic acid (48 per cent), m.p. 250°–252°. The identity of the acids prepared by the two methods was proved by a mixed melting point of methyl 6,7-methylenedioxy-2-phenylcinchoninate, m.p. 132°–133°.

1-Diethylamino-4-aminopentane was condensed with several derivatives of benzaldehyde to give a series of anils.<sup>14</sup> Two moles of acetic anhydride and phenothiazine with aluminum chloride in carbon disulfide gave (–), (–)-diacetylphenothiazine (18 per cent), m.p. 253°–254° and N-acetylphenothiazine (18 per cent). The acetyl groups in the diacetylphenothiazine could not be hydrolyzed in the usual manner, thus showing that neither group was on the nitrogen atom.

Dibutylamine and formic acid gave dibutylformamide (92 per cent), b.p. 76°–78° (0.25 mm.);  $n_D^{20}$  1.4435;  $d_4^{20}$  0.8932. Ethyl crotonate and N-bromosuccinimide in refluxing carbon tetrachloride yielded ethyl  $\gamma$ -bromocrotonate (82 per cent), b.p. 66°–67° (0.3 mm.);  $n_D^{16}$  1.492. 3-Diethylamino-2-hydroxybutyronitrile (62 per cent), b.p. 99°–100° (0.6 mm.), was prepared from 3-diethylamino-2-hydroxypropyl chloride, obtained from the condensation of epichlorohydrin with diethylamine, and potassium cyanide.

Complete physiological reports on these compounds are not available, but none of the compounds on which reports have been received has shown any significant therapeutic value.

<sup>13</sup> Private communication, Dr. R. C. Elderfield, Columbia University, New York, N. Y.

<sup>14</sup> Gilman and Massie, Jour. Amer. Chem. Soc., 68, 908 (1946).

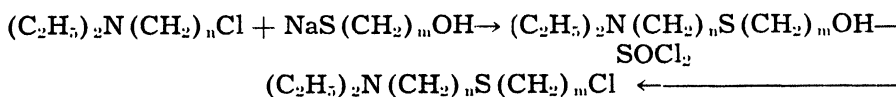
# FLUORINE AND SULFUR QUINOLINE DERIVATIVES AS ANTIMALARIAL AGENTS<sup>1</sup>

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A number of compounds that are closely related to known anti-malarial agents have been synthesized. The chief object of this investigation has been the correlation of chemical constitution with antimalarial activity.

A series of aliphatic diethylaminoalkyl chloroalkyl sulfides was prepared by the following reactions:



The compounds synthesized by this method were  $\gamma$ -diethylaminopropyl  $\gamma$ -hydroxypropyl sulfide (b.p. 126°–129°/0.1 mm.),  $\gamma$ -diethylaminopropyl  $\gamma$ -chloropropyl sulfide (b.p. 95°–97°/0.1 mm.;  $n_D^{20}$  1.4890;  $d_4^{20}$  0.9980),  $\gamma$ -diethylaminopropyl  $\beta$ -hydroxyethyl sulfide (b.p. 100°–102°/0.1 mm.), and  $\gamma$ -diethylaminopropyl  $\beta$ -chloroethyl sulfide (b.p. 71°–75°/0.1 mm.). These chlorosulfides were condensed with the appropriate aromatic amine to give the following compounds: 8-[ $\gamma$ -( $\gamma'$ -diethylaminopropylmercapto)-propylamino]-6-methoxyquinoline (b.p. 215°–220°/0.1 mm.), the dihydrochloride (m.p. 128°–130°); 8-[ $\beta$ -( $\gamma$ -diethylaminopropylmercapto)-ethylamino]-6-methoxyquinoline (b.p. 205°–210°/0.1 mm.), the dihydrochloride (m.p. 164°–165°); 3-[ $\gamma$ -( $\gamma'$ -diethylaminopropylmercapto)-propylamino]-trifluoromethylbenzene (b.p. 173°–175°/0.1 mm.), and the dihydrochloride (m.p. 123°–124°). The reaction of  $\gamma$ -chloropropyl bromide and sodium  $\beta$ -hydroxyethyl mercaptide gave an 85 per cent yield of  $\beta$ -hydroxyethyl  $\gamma$ -chloropropyl sulfide (b.p. 93°–96°/0.1 mm.,  $n_D^{20}$  1.5140,  $d_4^{20}$  1.1766). The acetylation of this hydroxysulfide gave  $\beta$ -acetoxyethyl  $\gamma$ -chloropropyl sulfide (b.p. 86°–87°/0.1 mm.  $n_D^{20}$  1.4879,  $d_4^{20}$  1.1531).

$\beta$ -Diethylaminoethyl mercaptan and  $\gamma$ -diethylaminopropyl mercaptan were prepared by the reaction of the corresponding chloride with aqueous sodium hydrosulfide. The following derivatives of these mercaptans were prepared:  $\beta$ -diethylaminoethyl mercaptan hydrochloride (m.p. 170°–172°),  $\beta$ -diethylaminoethyl 2,4-dinitrophenyl sulfide hydrochloride (m.p. 187°–188°), and  $\gamma$ -diethylaminopropyl 2,4-dinitrophenyl sulfide hydrochloride (m.p. 143°–145°).

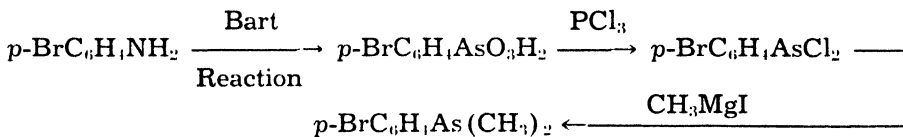
$\beta$ -Diethylaminoethyl  $\gamma$ -aminopropyl sulfide was prepared by the

<sup>1</sup> Doctoral thesis No. 779, submitted August 27, 1945.

Gabriel synthesis from  $\gamma$ -bromopropylphthalimide and  $\beta$ -diethylaminoethyl mercaptan. This aminosulfide was condensed with 6,9-dichloro-2-methoxyacridine in phenol to give 9-[ $\gamma$ -( $\beta$ -diethylaminoethylmercapto)-propylamino]-6-chloro-2-methoxyacridine (m.p.  $62^{\circ}$ – $64^{\circ}$ ), and the dihydrochloride (m.p.  $252^{\circ}$ – $254^{\circ}$ ). The condensation of  $\beta$ -diethylaminoethyl  $\gamma$ -aminopropyl sulfide and acetylacetone gave  $\beta$ -diethylaminoethyl  $\gamma$ -(2,5-dimethyl-1-pyreryl)-propyl sulfide.

The Skraup synthesis with 3-trifluoromethyl-4-nitroaniline gave 7-trifluoromethyl-6-nitroquinoline (m.p.  $164^{\circ}$ – $165^{\circ}$ ). The structure of this product was established by a stannous chloride reduction of the nitro compound to 7-trifluoromethyl-6-aminoquinoline (m.p.  $154^{\circ}$ – $155^{\circ}$ ), and then the deamination of this amine to give 7-trifluoromethylquinoline. The 7-trifluoromethylquinoline picrate (m.p.  $220^{\circ}$ – $221^{\circ}$ ) was compared with an authentic specimen (mixed m.p.). Acetylacetone and 7-trifluoromethyl-6-aminoquinoline gave 7-trifluoromethyl-6-(2,5-dimethyl-1-pyreryl)-quinoline (b.p.  $135^{\circ}$ – $138^{\circ}$ /1.0 mm., m.p.  $86^{\circ}$ – $87^{\circ}$ ); picrate (m.p.  $263^{\circ}$ – $265^{\circ}$ ). The condensation of 7-trifluoromethyl-6-aminoquinoline and  $\gamma$ -diethylaminopropyl chloride hydrochloride gave an oil which could not be purified by distillation, but a picrate (m.p.  $236^{\circ}$ – $238^{\circ}$ ) was prepared.

*p*-Bromophenyldimethylarsine was prepared in rather large quantities from *p*-bromoaniline by the following series of reactions:



*p*-Bromophenyldimethylarsine reacts readily with lithium to give the organolithium compound in good yield; the formation of the Grignard reagent from this bromide was slow, but the yield was good. *p*-Lithiophenyldimethylarsine was added to a series of quinoline derivatives to give the following compounds: dimethyl-*p*-(dihydro-2-quinolyphenyl)-arsine (b.p.  $185^{\circ}$ – $190^{\circ}$ /0.55 mm.); dimethyl-*p*-(2-quinolyphenyl)-arsine (m.p.  $65^{\circ}$ – $66^{\circ}$ ), the picrate (m.p.  $172^{\circ}$ – $173^{\circ}$ ); dimethyl-*p*-(8-methyl-2-quinolyphenyl)-arsine (b.p.  $129^{\circ}$ – $130^{\circ}$ /0.1 mm.), the picrate (m.p.  $129^{\circ}$ – $130^{\circ}$ ); dimethyl-*p*-(7-methyl-2-quinolyphenyl)-arsine (b.p.  $140^{\circ}$ – $145^{\circ}$ /0.15 mm.), the picrate (m.p.  $177^{\circ}$ – $177.5^{\circ}$ ); dimethyl-*p*-(6-chloro-2-quinolyphenyl)-arsine (m.p.  $123.5^{\circ}$ – $124^{\circ}$ ), the picrate (m.p.  $149^{\circ}$ – $150^{\circ}$ ); and dimethyl-*p*-(5,6-benzo-2-quinolyphenyl)-arsine (m.p.  $266^{\circ}$ – $168^{\circ}$ ), the picrate (m.p.  $176^{\circ}$ – $178^{\circ}$ ). The reaction with pyridine gave dimethyl-*p*-(2-pyridylphenyl)-arsine (b.p.  $155^{\circ}$ – $159^{\circ}$ /0.1 mm.); the picrate (m.p.  $150^{\circ}$ – $151^{\circ}$ ). The reaction with 3-cyanoquinoline gave a product which could not be purified. *p*-Dimethylarsinophenylmagnesium bromide and *p*-dimethylaminobenzaldehyde gave *p*-dimethylaminophenyl-*p'*-dimethylarsinophenylcarbinol (m.p.  $89^{\circ}$ – $90^{\circ}$ ).

The reaction of *p*-dimethylaminophenyllithium and 3-cyanoquinoline gave *p*-diethylaminophenyl 3-quinolyl ketone (b.p.  $210^{\circ}$ – $240^{\circ}$ /0.1 mm., m.p.  $147^{\circ}$ – $148^{\circ}$ ); the oxime (m.p.  $247^{\circ}$ – $249^{\circ}$ ).

Ethyl 3-quinolinecarboxylate was prepared from the acid using hydrogen chloride and absolute ethanol in 65 per cent yield. Methanolic hydrogen chloride and 3-cyanoquinoline gave methyl 3-quinolineimido-carboxylate (m.p. 195°–196°). The reaction of this imino ether with ammoniacal methanol failed to give the expected amidine. Ethyl 3-quinolinecarbonyl acetate (m.p. 85°–87°) was prepared from the condensation of ethyl 3-quinolinecarboxylate and ethyl acetate with sodium ethoxide. Treatment of this  $\beta$ -keto ester gave methyl 3-quinolyl ketone which was brominated to give bromomethyl 3-quinolyl ketone hydrobromide. The reaction of this bromide with 6-methoxy-8-aminoquinoline gave 6-methoxy-8-quinolylaminomethyl 3-quinolyl ketone (m.p. 268°–269°).

An attempt to prepare 3-aminoquinoline from 3-bromoquinoline and sodamide in liquid ammonia failed. 3-Aminoquinoline was prepared from 3-bromoquinoline, concentrated ammonium hydroxide, and copper sulfate in a steel bomb at 190°. A series of aminoquinoline derivatives was condensed with  $\gamma$ -diethylaminopropyl halide to give the following compounds: 3-( $\gamma$ -diethylaminopropylamino)-quinoline (b.p. 160°–195°/0.5 mm.), 5-( $\gamma$ -diethylaminopropylamino)-8-methylquinoline (b.p. 175°–195°/1.0 mm.), and 5-( $\gamma$ -diethylaminopropylamino)-6-methylquinoline (b.p. 160°–170°/1.0 mm.). The condensation of  $\gamma$ -diethylaminopropyl chloride and 3-amino-9-ethylcarbazole gave 3-( $\gamma$ -diethylaminopropylamino)-9-ethylcarbazole (b.p. 260°–265°/1.0 mm.). The reaction of  $\gamma$ -diethylaminopropyl chloride with carbazole at 200° in a sealed tube gave no product. This chloride and 9-carbazyllithium also failed to react.

*p*-Chlorophenylhydrazine was prepared and condensed with acetylacetone in acetic acid to give 1-(*p*-chloroanilino)-2,5-dimethylpyrrole (m.p. 88°–89°).

The results of the pharmacological tests, which were carried out under the auspices of the United States Government, are restricted. Accordingly, publication of such results must wait on future release.

# COAGULATION OF COLLOIDS BY ELECTROLYTES AS A FUNCTION OF CHARGE AND RELATIVE CONCENTRATIONS<sup>1</sup>

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Numerous investigations have been made of the relationship of the concentration of hydrophobic colloids to the coagulation value of an added electrolyte. Burton and Bishop<sup>2</sup> formulated a rule stating that the coagulation values of univalent ions increase with a decrease in sol concentration, of divalent ions are nearly independent of sol concentration, and of trivalent ions vary directly with the sol concentration. However, the hydrous oxide sols have been reported to be exceptions to this rule unless they were very pure.

The purpose of the present investigation was to study quantitatively the relationship of the Burton-Bishop rule to the purity of hydrous ferric oxide, a typical hydrous oxide. Some attention was also paid to the effect of aging and of the method of preparation of the colloid. Most of the study was based on the Sorum sol, prepared by dropping ferric chloride into boiling distilled water. The sols were purified by dialysis with cellophane tubing; the purities of the sols were calculated in terms of the ratio of the equivalents of iron to the equivalents of chloride. Data are given for the characteristics of the membrane on the basis of membrane thickness, specific water content, and rate of flow of water.

The course of agglomeration, defined as the collision and adhesion of colloidal particles, was followed by the change in transmission of light as measured by the KWSZ photometer. Coagulation was defined as that point of maximum agglomeration which ultimately would result in sedimentation. The time at which the direction of the change of light transmission reversed was taken as the time of coagulation and denoted as the *critical time* ( $t_c$ ). At the critical time, flocs were visible but sedimentation was not apparent.

The critical time, within limits, is an exponential function of the concentration of electrolyte; i.e., the logarithm of critical time is a linear function of the concentration of the electrolyte. This linear relation is found only for relatively small values of the critical time and is indicative of rapid coagulation. Critical times greater than about 45 minutes were indicative of slow coagulation.

In the coagulation of hydrous ferric oxide sols with electrolytes containing a dominating univalent ion, the straight lines, obtained by

<sup>1</sup> Doctoral thesis No. 795, submitted June 10, 1946.

<sup>2</sup> Burton, E. F., and Bishop, Miss E. 1920. Coagulation of colloidal solutions by electrolytes. Influence of the concentration of the sol. Jour. Phys. Chem. 24, 701-15.

plotting the logarithm of critical time against concentration of electrolyte, for different sol concentrations, intersect except in cases of highly purified sols, when the lines tend to be parallel. The slopes of the lines were determined from the relation

$$c = m \log t_c - b$$

where  $c$  is the concentration of the electrolyte. The intercepts of these lines with the  $y$ -axis are indicative of the order of the coagulation values for different sol concentrations up to the point of their intersection. The order of the slow coagulation values may be, but is not necessarily, the inverse of the rapid coagulation. With potassium and sodium chloride, the order of the rapid coagulation values, as indicated by the intercepts, increased with a decrease of the sol concentration except for a scrambling effect at purities of about 50 to 150. However, the order of slow coagulation values was in agreement with the Burton-Bishop relation only when the sol purity was 250 or higher.

The straight lines for different sol concentrations when potassium sulfate was employed are parallel, indicating that their order in rapid and slow coagulation is the same. An examination of the intercept values shows that there is a slight decrease of the coagulation values for low sol concentrations and a slight increase at higher concentrations. That is, the potassium sulfate has a slight tendency to behave as an electrolyte with a dominant univalent ion for low sol concentrations and as though it were trivalent for higher concentrations. The coagulation values thus are in accord with the Burton-Bishop rule indicating an intermediate position between the behavior of univalent and trivalent ions and a tendency toward being independent of the sol concentration.

Since potassium ferricyanide caused gelation as well as coagulation of the sol, a complete series of sols of different purities could not be investigated. Sufficient data were obtained, however, to show that the straight lines, obtained as before by plotting the logarithm of critical time and concentration of electrolyte, are parallel for different sol concentrations. The order of the intercept values indicated that the coagulation values of the electrolyte decreased with a decrease in sol concentration, which is in accord with the Burton-Bishop rule.

Further investigation should be made of the reason for the scrambling effect noted for certain purities of the sol when coagulated by sodium chloride and potassium chloride. Investigation also should be made of the slight tendency of potassium sulfate to show coagulation behavior typical both of univalent and of trivalent ions, depending on the concentration of the sol.

# A SOLUTION OF A RECTIFIER FILTER CIRCUIT WITH A CAPACITIVE INPUT<sup>1</sup>

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The full-wave rectifier circuit with a capacitive-input filter is used as the direct-current power supply in most radio receivers as well as in other electronic apparatus such as amplifiers, cathode-ray oscilloscopes, oscillators, and radar equipment. It is the purpose of this thesis to present an analysis of this rectifier circuit after certain simplifying assumptions have been made, and to exhibit various characteristics of the circuit in such a manner that they may be useful in the design of circuits of this type.

A résumé of the principles of the steady-state operational calculus was presented since this calculus was used to solve the circuit equations, and several theorems needed for the solution of rectifier circuits in particular were given. Certain assumptions were made to simplify the circuit equations sufficiently so that a solution was possible. The major assumptions were those of no resistance in the tube when conducting, no resistance in the filter inductor, and no leakage reactance and resistance in the transformer. The effect of these assumptions may be reduced to a considerable extent by a correction to be given later. Two equivalent circuits were used, one for the time when the tube is conducting and the other for the time of no conduction. From the solution it was found that all of the characteristics depended only on the two parameters, the ratio of the inductor reactance to capacitor reactance ( $\omega^2 LC$ ) and the ratio of load resistance to capacitor reactance ( $\omega CR$ ). Filter resonance occurred at  $\omega^2 LC = 0.5$ , and no calculations were made for  $\omega^2 LC < 0.6$  since this region has no practical importance. Two special cases of the circuit were calculated separately, and these are the case of  $\omega^2 LC$  infinitely large (infinitely large filter inductance) and the non-cut-off case (heavy loads). In the non-cut-off case the current flows continuously into the filter instead of flowing in pulses as is usual for lighter loads.

Three of the appendices at the end of the thesis presented the details of the derivation, method of calculation, and results for the general case and the two special cases mentioned above. The circuit equations were solved by use of the direct and inverse transforms and various theorems of the steady-state operational calculus. From the expressions for the steady-state currents and voltages obtained in this manner and from the fact that the tube current is zero when the tube is not conducting, five equations were found relating the angles, charges on the condensers, and currents in the inductor at the instants at which the tubes started and stopped conducting. The conditions occurring in the tube at the starting

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<sup>1</sup> Doctoral thesis No. 794, submitted May 31, 1946.



and stopping of conduction led to three more equations, giving eight equations in all involving the eight unknown angles, charges, and currents mentioned above. Since the equations were linear in the charges and currents, the elimination of these quantities was easily done, but the two remaining transcendental equations could not be solved explicitly for the two other unknowns, the starting and stopping angles of conduction. These angles were obtained by a graphical method of solution. The solution was made for a sufficient number of points so that graphical characteristics could be constructed from the results.

The characteristics that are most useful in the design of the circuit and in predetermining its properties are the tube angles, the output average voltage, the per cent ripple, the peak and average tube currents, and the peak inverse voltage on the tubes. These characteristics were presented as curves or equations varying with the two parameters,  $\omega^2 LC$  and  $\omega CR$ . The angle  $\gamma$  representing the length of time that each tube is conducting during a half cycle varied from zero degrees with an open-circuited load to 180 degrees at short circuit. The angle  $\beta$  which was the angle at which each tube stopped conducting with respect to the zero of the applied alternating voltage varied from 90 degrees at open circuit to 180 degrees at short circuit. The average output voltage ratio was defined as the ratio of the average output voltage to the maximum value of the input alternating voltage. This ratio was found to vary from unity at open circuit to 0.6366 at short circuit. The ratio in per cent of the effective ripple voltage to the average output voltage was used as the per cent ripple and varied from zero at open circuit to a maximum in the neighborhood of  $\omega CR$  equal to unity and then became zero again at short circuit. A partly empirical and partly theoretical equation for the per cent ripple was obtained and should prove very useful in design work. The peak tube current characteristic was presented as a ratio of the peak to average current in the tube and was found to vary from infinity at open circuit to two at short circuit. The average tube current was always one-half of the average output current, while the peak inverse voltage across each tube was always twice the maximum value of the input alternating voltage. The outline of a method for using these characteristics in the design of the rectifier and filter circuit was also presented.

The mathematical analysis had been preceded by some preliminary experimental work which was done to observe the operation of the circuit and to determine approximate values of parameters for use in the mathematical analysis. A more exact experimental study was made later to compare experimental with calculated results. By means of a special bridge circuit, an iron-core filter inductor was measured for its inductance and resistance with various alternating and direct currents passing through it. The capacitance and resistance of the filter condensers were measured by the use of another bridge circuit. A value for the resistance of the tubes used in the rectifier circuit was found from static voltage and current measurements made on the tubes. In the experimental test for the whole rectifier and filter circuit, data were taken from which were

obtained values of the angle  $\gamma$ , the average output voltage ratio, and the per cent ripple. These values were compared with the calculated results from the mathematical analysis and were found to agree very well except at quite heavy loads. By use of a simple correction involving the tube and filter-inductor resistance, the agreement even at heavy loads became very good.



## THE CYPERACEAE OF IOWA

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During the almost fifty years which have passed since R. I. Cratty published *The Iowa Sedges* (107)<sup>1</sup>, additional species of the family Cyperaceae have been reported from Iowa, and numerous notes on range extensions within the state have been published. Since these reports and notes have appeared in various publications, an evaluation of all that is now known about the sedges of our state has seemed desirable.

In 1871, Bessey (100), in what was probably the first publication to mention Iowa sedges<sup>2</sup>, listed 21 species. In 1876, Arthur (90) listed 51 species and 5 varieties, and during the next seven years (91, 92, 93, 94, 95) added 36 more names to the sedge flora of Iowa. Cratty, in 1898, citing all of the then available specimens, included 103 species and 11 varieties in *The Iowa Sedges* (107). In 1907, Wesley Green in *Plants of Iowa* (125), a compilation without locality records, mentioned 110 species and 11 varieties. He probably relied on Cratty for the list of sedges. In *The Iowa Flora* (114), written in 1932 but not published until 1933, Cratty admitted 120 species and 1 variety to the state list. The omission from that list of so common a species as *Cyperus strigosus* L., and its several varieties, is indeed strange. Recent papers by Hayden (128, 129, 130) supplement Cratty's state list.

In the present paper, 127 species and 10 varieties are recognized as comprising the known Cyperaceous flora of Iowa. Seven of the species are represented in the state only by varieties which are different from the typical phase of the species. Two species—*Carex bushii* Mackenz. and *Carex douglasii* Boott—are reported for the first time from Iowa, and the inclusion of *Scirpus torreyi* Olney among the Iowa citations in "Gray's" *Manual* (66) and Rydberg's *Flora* (67) is justified. Seven new varietal combinations are proposed.

In the preparation of this paper I have examined 3,727 specimens of Iowa sedges, most of them belonging to institutional or private herbaria located in Iowa. These specimens have been compared with the large number of specimens, from the entire range of each species, which were available in the herbarium of the New York Botanical Garden—where much of the work of preparing this paper was carried on during

<sup>1</sup> The numbers in parentheses refer to the papers or volumes listed in the Bibliography at the end of this paper.

<sup>2</sup> Torrey (80), in reporting on an 1837 collection of plants from the upper Mississippi Valley, does not mention Iowa localities for any of the 19 sedges included in his list. Under the date of 1848, Parry (64) cites many Iowa localities for other plants, but does not refer any of the 26 sedges in his list to our state. See also the discussion of this list by Ellsworth (116).

the years 1939 to 1941. I also have consulted the available literature on the flora of Iowa in an attempt to correlate scientific names in use at the time this literature was published with those accepted today. The opportunity to examine many of the specimens mentioned in the early literature has been exceedingly helpful in assuring the accuracy of such correlations.

The Iowa literature which contains references to sedges is included in the Literature Cited section of this paper as "Part II—Bibliography of Iowa Cyperaceae." A number of papers, which cite species of sedges from a particular geographical area or ecological habitat within the state but without mention of exact locality (140, 156, 160), are included. Part I of the Literature Cited includes the manuals, floras, and principal technical papers consulted in making nomenclatural decisions and preparing the statements on the total geographical distribution of species; a few such papers contain references to Iowa specimens of Cyperaceae.

Scientific names used in this paper do not agree entirely with those given in any one of the floras or manuals now in use in the Middle West. Insofar as it has been practical to do so, the nomenclature of this paper has been brought up to date. However, no attempt has been made to present a complete list of synonyms for each included species. Such a procedure, monographic in scope, would merely have added useless bulk. The majority of such synonyms as have been included are cited for either or both of two reasons: (a) when the name used for a species in either "Gray's" (66), Britton's (13, 14), or Rydberg's (67) manuals or in the *Illustrated Flora* (15, 16) differs from that used in this paper<sup>3</sup>, or (b) when a name used in earlier Iowa lists differs from that used here. In a few cases it also has been necessary to include synonyms relative to recent treatments of individual species. The common or vernacular names given for the species are essentially the same as those used in the *Illustrated Flora* (15, 16).

In the matter of recognition of species, I have followed more or less closely—but nevertheless conservatively—certain authorities on several genera of the family. The present treatment of *Carex* is essentially a modification of that of Mackenzie (53, 54), although Kükenthal (48) and a number of recent papers dealing with the species of this genus in neighboring states have been consulted (1, 20, 43, 44, 45, 81). Core (19) has been followed in *Scleria*, Beetle (4, 5, 6, 7) in *Scirpus*, and O'Neill (60) and co-workers—Corcoran (18), Horvat (46) and McGivney (56)—in *Cyperus*. The treatment of *Eleocharis* is based in part upon those of Fernald (23), Fernald and Brackett (35), and Svenson (71, 72, 73, 74, 76). In the remaining genera, for which no complete or recent monograph is available, it has been necessary to study and compare scattered notes on fragments of a genus.

<sup>3</sup> In such cases, initials in parentheses follow the synonym citation. Initials used for this purpose are: (B)—Britton's *Manual*; (BB)—*Illustrated Flora*; (BB2)—*Illustrated Flora*, ed. 2; (G)—"Gray's" *Manual*; (R)—Rydberg's *Flora*. See General Bibliography at end of this paper for exact bibliographic citation for these volumes.

Because the Cyperaceae are a morphologically specialized group of plants, a specialized technical vocabulary has been gradually developed as an aid to precise description and discussion of members of the family. Although every attempt has been made to simplify, as much as is practical, the terminology used in the identification keys, it has seemed impossible to eliminate entirely all of the special terms commonly used for this group. Indeed, the elimination of a single such term, which so precisely describes some detail of morphology, often necessitates the use of a long and devious descriptive clause in replacement. Therefore, in order to maintain the necessary clarity and precision in the keys, the more important terms have been retained; these are defined in the Glossary which immediately precedes the Bibliography. As a supplementary aid to the understanding of these terms, a series of simple illustrations (Fig. 3) has been prepared.

The specimens which I have examined in the course of this study are to be found in the following Iowa institutional herbaria: Coe College, Cedar Rapids (C); Grinnell College, Grinnell (GRC); Iowa State College, Ames (ISC); Iowa State Teachers College, Cedar Falls (T); Iowa Wesleyan College, Mt. Pleasant (IW); Parsons College, Fairfield (P); and State University of Iowa (SUI). I also have examined the specimens of Iowa Cyperaceae in the private herbaria of B. O. Wolden, of Estherville, Iowa (W); Dr. M. L. Grant, of Cedar Falls, Iowa (MLG); and Dr. J. D. Dwyer, of Albany, New York (D); and the Iowa specimens in the following eastern herbaria:<sup>4</sup> The Brooklyn Botanic Gardens, Brooklyn, N. Y. (B); the Gray Herbarium, Cambridge, Mass. (G); and the New York Botanical Garden, New York, N. Y. (NY).

Because of the large number of specimens examined during the preparation of this paper, it has seemed inadvisable to cite all of them at this time. To do so would more than double the size of this paper. Therefore, specimens have been cited only for the rare or the less well-known entities—34 species and 4 varieties out of the 127 species and 10 varieties in Iowa. The location of these specimens is indicated in the citation by the parenthetical letters given in the preceding paragraph. In addition, manuscript copies of the complete list of specimens examined are on file in the following libraries: Iowa State College, Ames, Iowa; State University of Iowa, Iowa City, Iowa; Missouri Botanical Garden, St. Louis, Missouri; New York Botanical Garden, New York, N. Y., and in the American Documentation Institute, 2101 Constitution Ave., Washington, D. C.

#### DISTRIBUTION AND GEOGRAPHICAL RELATIONSHIPS

Seventeen of the 137 entities (species and varieties) in the Iowa sedge flora are known, as yet, definitely only from one county. These, as

<sup>4</sup>Through the courtesy of a fellow student at the New York Botanical Garden, I also was able to examine the Iowa specimens of the genus *Hemicarpha* from the herbaria of the Chicago Natural History Museum (F) and the Missouri Botanical Garden (M).

a matter of minor interest, are: Allamakee County—*Carex tonsa*; Clinton County—*Scirpus torreyi*; Davis County—*Scirpus cyperinus* var. *rubricosus*; Emmet County—*Carex chordorrhiza*, *C. limosa*, *Eleocharis pauciflora* var. *fernaldii*, *E. wolfii*, and *Scleria verticillata*; Fayette County—*Carex conoidea*; Jasper County—*Carex douglasii* and *C. tuckermanni*; Jefferson County—*Carex crinita*; Muscatine County—*Bulbostylis capillaris* and *Fimbristylis autumnalis*; Palo Alto County—*Carex foenea* and *Scirpus paludosus*; Poweshiek County—*Carex richardsonii*. Five of these are known from only one or two early collections—*Carex conoidea* (in 1898), *C. douglasii* (in 1904 or 1905), *C. richardsonii* (in 1876 and 1879), *C. tuckermanni* (in 1886 and 1897), and *Scirpus torreyi* (in 1878)—and, dependent on the effects of agriculture on the original locality and habitat, may no longer be growing in Iowa. A careful and detailed investigation should be made to determine the status of these five species.

Distribution of the species within Iowa is shown on the accompanying small maps (Figs. 4-12), whereon each county in which a species is known to occur—on the basis of a specimen or specimens which I have examined—is indicated by a black dot, triangle, or cross. Unconfirmed county reports, those mentioned in the literature but from which I have not seen specimens, are included in the text. Casual examination of the maps might lead one to the conclusion that the greater proportion of the Iowa sedges are largely confined to the eastern and north-eastern one-third of the state, and that an area in the northwestern part of the state also contains a large number of species. It is true that the number of species decreases toward the western part of the state, and this decrease in number is correlated with a decrease of mesophytic habitats.<sup>5</sup> The reader should keep in mind, however, that comparatively little botanical work has been done in the south-central and southwestern portions of our state. Therefore, the maps for many of the Iowa species merely show where a particular species has been collected rather than where it actually grows.

For the convenience of the reader who might wish for some points of geographical, geological, and vegetational reference when considering the possible significance of species distributions as shown on the 114 maps of Figures 4 to 12, inclusive, the following seven Iowa maps have been included in this paper (Fig. 1, A—G): A. a key map to the counties of Iowa; B. a generalized topographical map; C. a map showing the boundaries of the glacial drift sheets; D. a map of soil types; E. a map indicating the major drainage basins; F. a map showing average annual precipitation and average length of frost-free growing season; and G. a map indicating the extent of the forest and prairie in the state at the time the white man came to Iowa.

<sup>5</sup> Because of the excellent consideration of general plant ecology and plant distribution in Iowa recently published by Hayden (129), further elaboration on this theme seems superfluous here. Additional references on the subject will be found in Dr. Hayden's list of Pertinent Literature. A number of papers, cited elsewhere in the present paper, should be consulted also; these are by W. A. Anderson (89), Pammel (136, 137, 141, 142), Shimek (154, 156, 157, 158, 159, 160), Wolden (169), and Wylie (171).

FIGURE 1. Reference Maps (See page 61)

A—County Key Map: Counties may be identified by means of corresponding numbers on map, as follows:

Adair .....	71	Jefferson .....	87
Adams .....	81	Johnson .....	65
Allamakee .....	15	Jones .....	53
Appanoose .....	96	Keokuk .....	76
Audubon .....	58	Kossuth .....	9
Benton .....	51	Lee .....	99
Black Hawk .....	39	Linn .....	52
Boone .....	47	Louisa .....	78
Bremer .....	23	Lucas .....	84
Buchanan .....	40	Lyon .....	1
Buena Vista .....	22	Madison .....	72
Butler .....	27	Mahaska .....	75
Calhoun .....	34	Marion .....	74
Carroll .....	45	Marshall .....	49
Cass .....	70	Mills .....	79
Cedar .....	66	Mitchell .....	12
Cerro Gordo .....	17	Monona .....	43
Cherokee .....	21	Monroe .....	85
Chickasaw .....	19	Montgomery .....	80
Clarke .....	83	Muscatine .....	68
Clay .....	7	O'Brien .....	6
Clayton .....	30	Osceola .....	2
Clinton .....	55	Page .....	91
Crawford .....	44	Palo Alto .....	8
Dallas .....	60	Plymouth .....	20
Davis .....	97	Pocahontas .....	23
Decatur .....	94	Polk .....	61
Delaware .....	41	Pottawattamie .....	69
Des Moines .....	89	Poweshiek .....	63
Dickinson .....	3	Ringgold .....	93
Dubuque .....	42	Sac .....	33
Emmet .....	4	Scott .....	67
Fayette .....	29	Shelby .....	57
Floyd .....	18	Sioux .....	5
Franklin .....	26	Story .....	48
Fremont .....	90	Tama .....	50
Greene .....	46	Taylor .....	92
Grundy .....	38	Union .....	82
Guthrie .....	59	Van Buren .....	98
Hamilton .....	36	Wapello .....	86
Hancock .....	16	Warren .....	73
Hardin .....	37	Washington .....	77
Harrison .....	56	Wayne .....	95
Henry .....	88	Webster .....	35
Howard .....	13	Winnebago .....	10
Humboldt .....	24	Winneshiek .....	14
Ida .....	32	Woodbury .....	31
Iowa .....	64	Worth .....	11
Jackson .....	54	Wright .....	25
Jasper .....	62		



FIG. 1, B.—Topography: The contours—generalized from map published by Lees (52a)—are shown in intervals of 200 feet. Elevations, in feet, between contour lines are as follows: 1—under 600; 2—600 to 800; 3—800 to 1,000; 4—1,000 to 1,200; 5—1,200 to 1,400; 6—1,400 to 1,600; 7—over 1,600. The extremes of altitude in Iowa are 448 feet at Keokuk, in the southeast and 1,670 feet on Ocheyedan Mound (Osceola County) in the northwest.

FIG. 1, C.—Glaciation: Boundaries of the several drift sheets, together with the location of a glacial lake and indication of the major glacial outwash areas. The drift sheets are numbered in sequence from the oldest to the most recent one: 1a—Nebraskan; 1b—Nebraskan exposed in stream valleys, Kansan evident along hill tops; 2—Kansan; 3—Illinoian; 4—Iowan substage of the Wisconsin (on many maps, and by many authorities, this is considered as a separate period of glaciation, "*the Iowan*"); 5—Mankato substage of the Wisconsin; 6—the approximate boundaries of the glacial Lake Calvin, of Illinoian age; 7—major areas of glacial outwash deposits. Drift sheet, lake, and outwash boundaries have been generalized from maps published by Kay and Graham (48b) and Flint (36a).

FIG. 1, D.—Soil Types: Boundaries of the major soil groups, as shown on the small map included in most of the Iowa county soil survey pamphlets, have been here correlated with the glacial drift sheet boundaries as given in map C of this figure. The correlated soil type areas may be listed as follows: 1—Nebraskan till; 2—Mississippi loess; 3—south Iowa loess over Kansan till; 4—Illinoian till remnants; 5—Kansan and Nebraskan till; 6—Missouri loess; 7—Iowan till; 8—Wisconsin till.

FIG. 1, E.—Drainage Patterns: The heavy continuous line X—X delimits the tributaries of the Missouri River (to the west and south) from those of the Mississippi River (to the east); the heavy continuous line Z—Z delimits a tributary of the Minnesota River (to the north) from the tributaries of the Mississippi River (to the south and east). Drainage basin units are defined as follows: 1—Upper Iowa and Yellow Rivers; 2a—Turkey and Maquoketa Rivers; 2b—Wapsipinicon River; 3a—Cedar River; 3b—Iowa River; 4—Skunk (or Chicaqua) River; 5—Des Moines River and tributaries; 6a—Chariton River; 6b—West Grand and East Grand Rivers; 7a—Nodaway and Little Platte Rivers; 7b—Nishnabotna and Tarkio Rivers; 8—Boyer River; 9—Little Sioux River; 10—Rock and Floyd Rivers; 11—Blue Earth River. The map has been simplified from one published by Kay and Apfel (48a).

FIG. 1, F.—Climatic Areas: The heavy continuous lines are iso-hyets which connect localities of equal average annual precipitation, in inches, as follows: a—26; b—28; c—30; d—32; e—34; f—36. The shading represents areas of equal average frost-free growing season, in terms of days, as follows: 1—130 to 140; 2—140 to 150; 3—150 to 160; 4—160 to 170; 5—170 to 180. This map has been adapted from two maps published by Reed (65a).

FIG. 1, G.—Original Vegetation: The distribution of Prairie and Forest (or Woodland) based on the original township surveys of 1832 to 1859. 1—areas of predominant heavy forest with little, if any, grassland areas on the higher ground; 2—heavy to moderately heavy forest along and near streams and bushland or grassland on the higher ground; 3—scattered to light or moderate forest along streams and large areas of grassland on the higher ground between the streams; 4—predominantly prairie. This map has been generalized from a map prepared under the direction of the Iowa State Planning Board and since published in several places, including the recent paper by Hayden (129). It should be noted that, because of the small scale of the map presented here, the width of the forest strips along streams is considerably exaggerated.

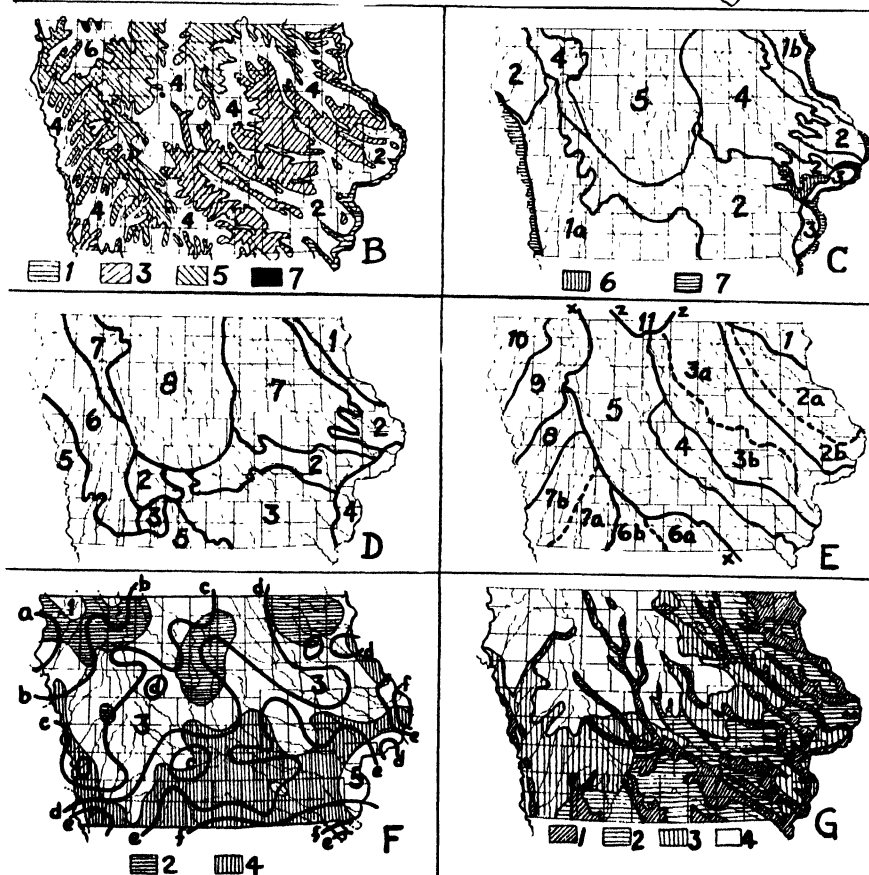
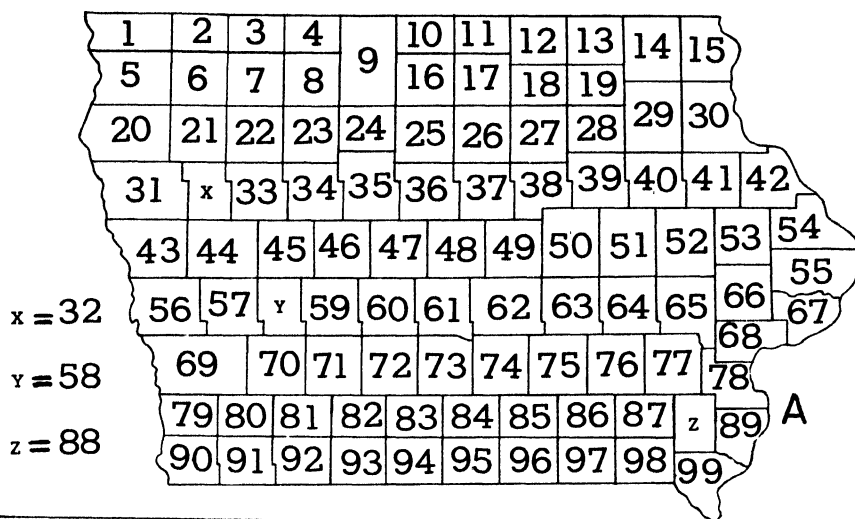


FIG. 1. Reference Maps.

The larger map of the state (Fig. 2-A) shows graphically the number of species of sedges now known from each county in Iowa. It will be noted that the Cyperaceous flora is fairly well known in only nine counties: Emmet, with 82 species; Webster, with 64; Johnson, with 60; Story, with 56; Clay, with 55; Palo Alto and Jefferson, each with 52 species; Lee and Poweshiek, each with 41. I have seen no specimens of sedges from Adams, Audubon, Cedar, Ida, Monroe, and Pocahontas Counties, although one or more species have been reported for each<sup>6</sup>. It is hoped that this preliminary treatment of the Iowa Cyperaceae may stimulate at least some of those who read it to make collections of the family in areas that are poorly represented by herbarium specimens. Thus, at a later date, it may become possible to prepare a more accurate and complete picture of the sedge flora of our state.<sup>7</sup>

Cratty (107) presented a comparative tabulation of the number of species in each genus of the Cyperaceae known at that time (1898) to occur in the seven states neighboring Iowa. I can see no particular advantage in amplifying such a tabulation in this paper but, for the purpose of a general comparison, the inclusion of a map (Fig. 2-B) indicating the total number of species in the family known from each of the thirteen states of the upper Mississippi valley seems to be in order.<sup>8</sup> The decrease in number of species toward the western part of this area, as with the decrease in number from east to west in Iowa, seems to be in direct relationship to the decrease in available moisture and decrease in diversity of habitat types. The considerable difference between the total for Missouri and that for Iowa is due, in part, to the presence in the former state of many more coastal plain species, as well as more species with a southwestern center of distribution. It should be noted, also, that the comparative smallness of the Iowa total is in part a result of the somewhat conservative delimitation of species in the present paper. An additional 22 "species" would be added to the Iowa total if the majority of recent treatments of some of the Cyperaceous genera had been strictly followed. The low total for Kentucky is largely due to the lack of botanical collecting in much of that state.

The Cyperaceous flora of Iowa, if considered from the standpoint of

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<sup>6</sup> At the time this paper was nearly completed, I had seen no specimens of Cyperaceae from either Cass County or Montgomery County, and had been unable to find a report of any species from the former. An unexpected opportunity to make a hurried trip into the southwestern corner of the state presented the opportunity of collecting 7 species in Cass County and 1 in Montgomery County. These were collected in wet ditches along the highway.

<sup>7</sup> I will deem it a pleasure to receive specimens of this family from Iowa for identification, or for confirmation of identification. Such specimens will, with the senders' permission, be placed in the herbarium of the Iowa State College Department of Botany. They also will be used by members of the Committee on Biological Survey of the Iowa Academy of Science in preparation of a state flora. Iowa specimens of families other than the Cyperaceae also are solicited.

<sup>8</sup> Not all of the state totals, as given on the map, are absolutely complete, but they will serve for a general comparison with the Iowa total. These totals were compiled from various publications (2, 3, 9, 21, 39, 43, 44, 45, 47, 48, 51, 52, 55, 58, 59, 61, 62, 63, 65, 68, 69, 70, 77, 78, 82, 83, 84, 85, and 86, among others) and from the specimens available at the New York Botanical Garden in 1941.



the center of distribution and the total distributional range of each of the species, may be divided into geographical-affinity groups as follows:

1. *Western*—species with center of distribution in the Great Basin, the Rocky Mountains, or the Western Plains; the eastern distributional margins reached for most of these species in Iowa (8 species, 6.3 per cent of the state total).

2. *Interior*—essentially prairie species which may or may not have an eastward extension of range along the old "prairie peninsula"; a few of these species seem to have Ozarkian affinities (33 species, 26.0 per cent of the state total).

3. *Coastal Plain—Embayment*—species ranging to a greater or lesser degree along the Atlantic and Gulf coastal plains and extending up the Mississippi valley (5 species, 4.0 per cent of the state total).

4. *Tropical American*—species with North American distribution similar to those in Group 3, but with the center of distribution in the tropics rather than in the United States (5 species, 4.0 per cent of the state total).

5. *Eastern*—species with a wide distribution through eastern North America, usually from Maine and southern Canada to the Gulf coast, and from the Atlantic coast to the margin between the prairies and plains. The majority of these species reach their western distributional limits in eastern Texas, Kansas, Nebraska, and western Minnesota; four of these species also have a disjunct distributional area on the Pacific coast (22 species, 17.3 per cent of the state total).

6. *Northeastern Woodland*—species essentially woodland in habitat preference, with their centers of distribution in the eastern Great Lakes area, their ranges sometimes extending southward in the Appalachian Mountains and reaching a narrowing western limit of distribution in the upper Mississippi Valley (20 species, 15.7 per cent of the state total).

7. *American Northern and Sub-Boreal*—species ranging from Newfoundland and Labrador and the Hudson Bay region, with their southern limits in the New England States, the Northern Lake States and northern Iowa, with or without a westward extension of range to the Pacific Coast and Alaska and a southward extension of range in the higher Rocky Mountains (22 species, 17.3 per cent of the state total).

8. *Eurasian—American Sub-Boreal*—species with the American portion of their ranges much as described for Group 7, but also ranging more or less throughout northern Europe and Asia (6 species, 4.7 per cent of state total).

9. *Temperate Weedy*—species widely distributed through North America and in the temperate regions of the other continents (6 species, 4.7 per cent of the state total).

#### ACKNOWLEDGMENTS

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For their kindness in allowing me to examine specimens from their institutional or private herbaria, and for additional facts and helpful suggestions, I express my thanks to: Dr. W. A. Anderson, of the State University of Iowa Botany Department; Dr. A. A. Beetle, of the Agricultural Experiment Station of the University of California; the late Dr. Charles Carter, of the Parsons College Biology Department; Dr. H. S. Conard, of the Grinnell College Botany Department; Dr. J. D. Dwyer, of the Albany College of Pharmacy, Albany, N. Y.; Dr. M. L. Grant, of the Iowa State Teachers College Science Department; Dr. Ada Hayden, of the Iowa State College Botany Department; Dr. H. E. Jaques, of the Iowa Wesleyan College Biology Department; Dr. H. K. Svenson, of the Brooklyn Botanic Gardens; Dr. C. A. Weatherby, of the Gray Herbarium; Drs. L. R. Wilson and W. N. Keck, of the Coe College Biology Department; and B. O. Wolden, of Estherville, Iowa.

#### CYPERACEAE (SEDGE FAMILY)—SYSTEMATIC TREATMENT

Annual or perennial grass-like or rush-like plants with slender, solid (rarely hollow) and usually triangular (sometimes terete or flattened) culms. The roots of most annual species are fibrous, while those of the perennial species are rhizomatous. The leaves are normally 3-ranked, narrow and grass-like, and with tubular, closed sheaths (Fig. 3-O); occasionally the leaves are reduced to bladeless sheaths (Fig. 3-U). A grass-like ligule is present in *Carex*. The flowers are perfect or unisexual and arranged in 1- to many-flowered spikelets or spikes (Fig. 3-A, B, C), each flower subtended by one or more scales (bractlets); the spikelets are solitary, or several together, or numerous and more or less clustered. The scales of the spikelets are either 2-ranked (Fig. 3-A) or spirally imbricated (Fig. 3-B) on the rachillae of the spikelets. The perianth is sometimes lacking; when present it is rudimentary, hypogynous, and composed of either bristles (Fig. 3-N, J), scales (Fig. 3-K), or scales and bristles (Fig. 3-I). In *Scleria*, the pistillate flower, and the resultant achene, are attached inside of a hard basal cup (the hypogynium; Fig. 3-V). In *Carex*, the pistillate flower, and the resultant achene, are enclosed in a perigynium (Fig. 3-P, Q, S), a more or less inflated sack-like structure which is a modified leaf sheath. Stamens 1 to 3, or rarely more; their filaments slender or filiform, the anthers 2-celled and basally attached. The ovary, although composed of 2 or 3 (or, rarely, 4 or more) united carpels, is 1-celled and 1-ovuled; the ovule is basally attached and anatropous. The style is usually 2- or 3-cleft. The fruit is a lenticular, plano-convex, trigonous, or nearly terete achene (Fig. 3-M); the achene is usually hard-walled (in *Scleria*, bony). The endosperm of the seed is mealy and the embryo very minute.

This family is world-wide in its distribution; it is usually subdivided into about 70 genera, under which approximately 3,000 species are recognized as distinct.

Sedges, for the most part, are plants of damp or wet places. Sloughs, swamps, bogs, marshes, ditches, and mud flats along river, pond or lake

FIGURE 3. Illustrations of Descriptive Terminology Used in This Paper.

- A—spikelet with two-ranked scales.
- B—spikelet with spirally imbricated scales.
- C—an achene of *Scleria* showing the several scales which subtend the single flower.
- D—pistillate flower of *Carex*, here shown with three stigmas.
- E—staminate flower of *Carex*.
- F—perfect flower of *Cyperus*, *Scirpus*, etc., here shown with two stigmas.
- G—achene of *Eleocharis* showing the apical tubercle formed from persistent style-base tissue and lack of perianth bristles; other species have perianth bristles of the type shown in H.
- H—achene of *Scirpus* showing terminal apiculation and simple perianth bristles.
- I—achene of *Fuirena*, with style still attached, showing perianth composed of alternating bristles and stipitate scales.
- J—achene of *Eriophorum* showing numerous capillary and silky perianth bristles.
- K—achene of *Hemicarpha* showing perianth scale between achene and rachilla of spikelet.
- L—diagrams of spike, spikelet, and scale shapes.
  - L-1—linear.
  - L-2—oblong.
  - L-3—lanceolate.
  - L-4—ovate.
  - L-5—elliptic.
  - L-6—oblancoate.
  - L-7—obovate.
  - L-8—orbicular.
- M—diagrams of achene or perigynium cross-sectional shapes.
  - M-1—lenticular or biconvex.
  - M-2—plano-convex.
  - M-3—triangular.
  - M-4—terete.
- N—diagrams of scale apex types.
  - N-1—truncate.
  - N-2—rounded or obtuse.
  - N-3—acute.
  - N-4—mucronate.
  - N-5—acuminate.
  - N-6—aristate or awned.
  - N-7—bifid and aristate.
- O—lateral view of leaf sheath (*dor*—dorsal surface; *ven*—ventral surface; *cu*—culm; *bl*—leaf blade).
- P—lateral view of a *Carex* perigynium (*dor*—dorsal surface; *ven*—ventral surface; *sc*—subtending scale; *bk*—beak of perigynium).
- Q—dorsal view of a wing-margined or bi-keeled perigynium with a bidentate beak.
- R—diagrammatic cross-section of such a wing-margined perigynium.
- S—dorsal view of a beakless, equal-nerved and nonmargined perigynium.
- T—diagrammatic cross-section of such an equal-nerved perigynium.
- U—bladeless basal leaf sheaths, characteristic of *Eleocharis*.
- V—achene of *Scleria*, showing hypogynium at its base.
- W—Lateral view of a *Carex* perigynium with an entire, obliquely-cut tubular beak.
- X—apex of leaf sheath (*sh*) and base of leaf blade (*bl*) of *Carex* showing position of ligule (*lg*) around culm (*cu*).

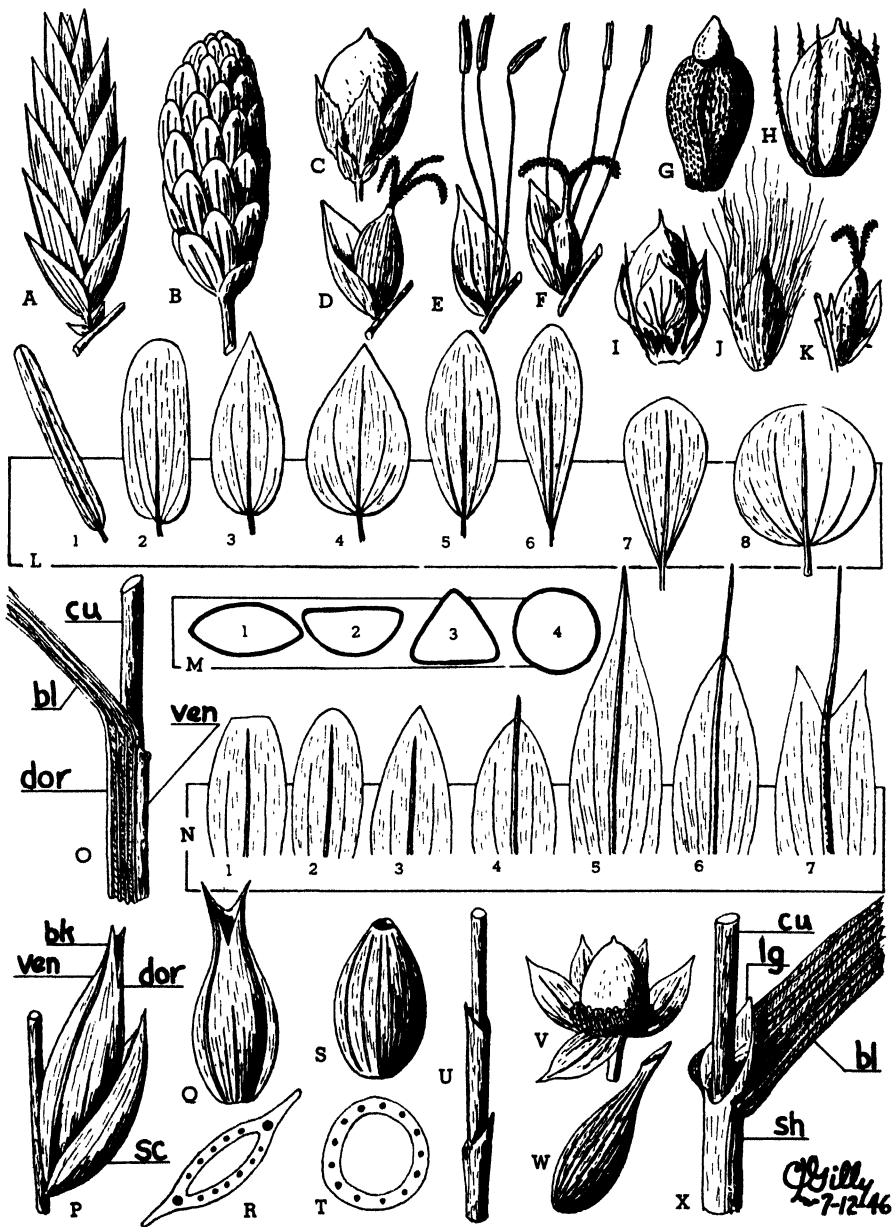


FIG. 3. Illustrations of Descriptive Terminology.



edges are among the preferred habitats. Some of the moisture-loving species occasionally are found in shallow or even deep water in ponds, lakes or streams, and a few species are wholly aquatic. Some species, particularly in the genus *Carex*, are inhabitants of moist or dry woodlands. Some few species are found on dry plains and in sandy shore and dune areas, and others on rocky ledges or in cliff crevices. This group of plants is indirectly of considerable economic importance. Many species play an important role in preventing soil erosion. Others play an equally important part in the building of soil along pond and lake shores. A few species may, on occasion, be troublesome weeds.

In studying the sedges it is necessary, because of the small size of the flowers, subtending bracts, and achenes, to use a hand lens of at least medium power. A 10x lens is reasonably satisfactory for observing most details, but a 15x or 20x lens is preferable. Another fact of utmost importance to remember in studying and identifying Cyperaceae is the necessity of having specimens with mature achenes. This is particularly true for the person who is making his first acquaintance of the group, and even the experienced botanist—unless he has a large collection of authentic specimens available for comparison—will encounter considerable difficulty in trying to identify immature specimens of sedges. *Eleocharis* and some groups of *Carex* present the most trouble in this respect. Such difficulty is the combined result of the comparatively simple structure of the flower in this family, and the fact that the principle differences between species usually are to be found in characters of the achene, subtending scales, and (in *Carex*) the perigynia.

#### KEY TO THE IOWA GENERA

- A. Culms jointed, leafy to the top; leaves cauline, one at each node; the inflorescence composed of several axillary clusters of spikelets; the scales of the spikelets 2-ranked (Fig. 3-A) ..... 1. *DULICHIMUM*
- AA. Culms not jointed (or the joints so short that all the leaves appear to be basal); inflorescence terminal or subterminal.
  - B. Each flower subtended by 1 to several scales, but the pistillate flower (and the resultant achene) of monoecious or dioecious plants not enclosed in a sack-like perigynium.
    - C. Scales of the more or less flattened spikelets 2-ranked (Fig. 3-A) ..... 2. *CYPERUS*
  - CC. Scales of the essentially terete spikelets spirally imbricated (Fig. 3-B).
    - D. Flowers aggregated in spikelets, each flower subtended by a single scale (Fig. 3-D, E, F); flowers all, or at least some in each spikelet, perfect; achenes not bony and shining white.
      - E. Style completely deciduous from achene at maturity, the achene blunt or merely apiculate at apex (Fig. 3-H, I, J).
      - F. Spikes numerous-flowered, the flowers all perfect (Fig. 3-F).
        - G. Perianth composed of 3 flattened and more or less stipitate scales alternating with 3 bristles (Fig. 3-I) ..... (*FUIRENA*)
        - GG. Perianth composed wholly of bristles (Fig. 3-H, J), or of a single minute scale (Fig. 3-K), or absent.

\* *Fuirena* and *Cladium* have been reported from Iowa, but as yet I have not been able to find Iowa specimens of either genus; the two genera are included in this generic key on the bare chance that they may yet be found, or rediscovered, within the state. See also "Genera Frequently Reported for Iowa," page 134 of this paper.

- H. Plants small, rarely more than 3 cm. tall; culms and leaves filiform; inflorescence of 1 to several sessile spikelets, subtended by a usually solitary slender bract; perianth of a minute scale between achene and spikelet rachis (Fig. 3-K; this scale sometimes absent); stigmas 2 ..... 3. *HEMICARPHA*
- HH. Plants taller and without the above combination of characters; perianth composed of bristles or of silky hairs (Fig. 3-H, J), or absent; stigmas 3 or 2.
  - I. Style not at all inflated at base; perianth bristles usually present (or, when absent, the achenes not minutely reticulate and white).
    - J. Perianth bristles 1-6 (Fig. 3-H) and hidden by the scales (in one species slightly exceeding the scales and thus visible), or sometimes absent ..... 4. *SCIRPUS*
    - JJ. Perianth bristles numerous, long and silky (Fig. 3-J), at maturity 4 mm.-20 mm. longer than the scales ..... 5. *ERIOPHORUM*
  - II. Style inflated and enlarged at base; perianth bristles absent; achenes minutely reticulate, white ..... 6. *FIMBRISTYLIS*
- FF. Spikelets few-flowered, the lower 1 or 2 flowers staminate, the upper 1 or 2 perfect ..... (CLADIUM)<sup>a</sup>
- EE. Style base persistent as a tubercle on summit of achene (Fig. 3-G).
  - F. Spikelets numerous-flowered, all flowers perfect.
    - G. Leaves present at the base of the culm; spikelets numerous in terminal umbels, subtended by 1-3, narrow, leaf-like involucre bracts ..... 7. *BULBOSTYLIS*
    - GG. Leaves reduced to bladeless sheaths (Fig. 3-U); spikes solitary, terminal, not subtended by leaf-like involucre bracts ..... 8. *ELEOCHARIS*
  - FF. Spikelets few-flowered, the upper 1 or 2 flowers staminate, the lower 1 or 2 perfect ..... 9. *RHYNCHOSPORA*
- DD. Pistillate flowers usually solitary, each surrounded by several scales (Fig. 3-C); flowers all unisexual; achenes bony, white and shining (Fig. 3-V) ..... 10. *SCLERIA*
- BB. Each flower subtended by 1 scale; flowers unisexual; each pistillate flower (and the resultant achene) enclosed in a sack-like perigynium (Fig. 3-P, Q, S) ..... 11. *CAREX*

# 1. DULICHIMUM Pers. Syn. 1: 65. 1805.

A monotypic genus, its single species confined to North America. Probably the most distinctive sedge in Iowa.

1. *Dulichium arundinaceum* (L.) Britt. Bul. Torrey Club 21:29. 1894.  
*Cyperus arundinaceus* L. Sp. Pl. 44. 1753.  
*D. spathaceum* (L.) Pers. Syn. 1:65. 1805.

*Dulichium*—usually found in boggy or swampy spots or along marshy shores—Newfoundland to northern Ontario, Minnesota and Nebraska, south to northern Florida, Louisiana, and Texas; also in northwestern Montana, northern Idaho and along the Pacific coast from southern British Columbia to extreme northern California.

IOWA DISTRIBUTION: map 4-a. SPECIMENS EXAMINED: Henry County, August 8, 1899, *Savage* (SUI). Jasper County, Lynnville, July 3, 1886, *Norris* (GRC). Winnebago County, August, 1930, *Pammel* (ISC); Forest City, July 4, 1921, *Pammel* (ISC). Worth County, Fertile, September, 1908, *Pammel* (ISC). UNCONFIRMED COUNTY REPORTS: Cerro Gordo (142), Emmett (142), Hamilton (142), Muscatine (99, 107), Scott (99, 107), Story (107), and Wright (142).

## 2. CYPERUS L. Sp. Pl. 44. 1753.

A large genus, in which about 650 species are recognized, with a world-wide distribution centered in the tropics and sub-tropics. Eleven species have been found in Iowa.

## KEY TO THE IOWA SPECIES

- A. Each spikelet composed of several to numerous flowers (and resultant achenes); each flower (and achene) subtended by a scale.
- B. Rachilla wings, when present, thin and transparent, not clasping the achenes; rachillae of the spikelets persistent on the rachis, or the entire spikelets falling as units.
- C. Styles 3-parted; achenes essentially triangular.
  - D. Scales tipped with a prominent recurved awn..... 1. *C. aristatus*
  - DD. Scales obtuse, acute or mucronate, the tips neither conspicuously awned nor recurved.
  - E. Spikelets about twice as wide as long; scales wide-spreading, the lowermost much longer than the upper ones on the same spikelet (each spikelet thus appearing to be ovate or triangular in shape) ..... 2. *C. acuminatus*
  - EE. Spikelets linear, at least several times longer than wide; scales all approximately equal in length and more or less appressed.
  - F. Inflorescence a compound umbel, the spikelets pinnately disposed on each rachis.
  - G. Rachilla wings, when present, permanently adnate to the rachillae; achenes 1.5 mm. or more in length.
  - H. Achenes sharply triangular, ellipsoid, scarcely more than twice as long as thick and almost as long as the subtending scales; scales ovate, more or less rounded or obtuse at the apices, slightly spreading, deciduous.
  - I. Leaf margins and culms smooth; spikelets 1.5 mm. or less in width; scales acute, shining brown or golden-brown 3. *C. esculentus*
  - II. Leaf margins and culms scabrous (or the culms rarely smooth); spikelets 2.5 mm.-5 mm. in width; scales mucronate, dull green or greenish ..... 4. *C. schweinitzii*
  - HH. Achenes obtusely triangular, at least four times as long as thick, less than one-half as long as the subtending scales; scales lanceolate, acute, closely appressed, persistent on the rachillae ..... 5. *C. strigosus*
  - GG. Rachilla-wings separating from the rachillae as pairs of basally attached scales; achenes 0.5 mm.-0.8 mm. long; scales bright reddish-brown ..... 6. *C. erythrorhizos*
  - FF. Inflorescence a solitary, sessile capitate cluster of spikelets, or when (rarely) compound, the spikelets capitate clustered at the apex of each rachis ..... 7. *C. filiculmis*
  - CC. Styles 2-parted; achenes lenticular or plano-convex.
    - D. Stigmas conspicuously exserted beyond the glumes, the spikelets thus superficially appearing to be ciliate; scales thin-textured, dull; achenes olive-grey ..... 8. *C. diandrus*
    - DD. Stigmas not, or scarcely, exserted beyond the scales, the spikelets thus appearing glabrous; scales firm, shining; achenes yellowish or brown ..... 9. *C. rivularis*
  - BB. Rachilla wings thick-textured, not transparent, incurved and clasping the achene; spikelets breaking between the scales into 1-fruited joints; styles 3-parted, achenes more or less triangular.
  - C. Scales closely imbricated, overlapping the next above on the same side of the rachilla ..... 10. *C. odoratus*  
var. *squarrosus*
  - CC. Scales distant, scarcely reaching to the middle of the next above on the same side of the rachilla ..... 11. *C. engelmanni*
  - AA. Spikelets, although 1-flowered and with only 1 achene each, bearing several empty scales and crowded into dense globular sessile heads; style 2-parted; achenes lenticular ..... (*C. tenuifolius*; see page 76)

1. *Cyperus aristatus* Rottb. Descr. and Icon. 23. 1773.*C. inflexus* Muhl. Gram. 16. 1817 (B, BB, BB2, R).*C. aristatus* var. *inflexus* (Muhl.) Kük. Pflanzenr. 101[IV. 20]: 504. 1936.

Awed cyperus—usually found along muddy streams or pond banks, sometimes in sandy soil—New Brunswick to Manitoba and British Columbia, south to Florida, Texas, northern Mexico and California; widely distributed in the tropics.

IOWA DISTRIBUTION: map 4-b. UNCONFIRMED COUNTY REPORTS: Cerro Gordo (107, 142), Hardin (148), Muscatine (99, 107, 159), and Scott (99, 107).

2. *Cyperus acuminatus* Torr. & Hook. Ann. Lyc. N. Y. 3: 435. 1836.

Acuminate cyperus—usually in wet soil in a variety of habitats—Indiana to Iowa, Kansas, Colorado, and Oregon, south to Louisiana, Texas, and California.

IOWA DISTRIBUTION: map 4-c. SPECIMENS EXAMINED: Hamilton County, 1902, *Pammel* (ISC). Jefferson County, Collett, 1897, *Baldwin* (G, ISC). Lee County, without date, *Fults* 1535 (ISC). Scott County, July 8, 1896, *Barnes and Miller* (ISC). Van Buren County, Keosauqua, summer of 1933, *Fults* (ISC). Woodbury County, Sioux City, without date, *Hitchcock* (SUI). UNCONFIRMED COUNTY REPORTS: Clinton (99, 107), Harrison (159), and Plymouth (93, 107).

3. *Cyperus esculentus* L. Sp. Pl. 45. 1753.*C. phymatodes* Muhl. Gram. 23. 1817.

Yellow nut-grass—usually in low, wet ground; spreading, apparently, by both seed and tuber-bearing root-stocks, this species frequently becomes a somewhat troublesome pest in cultivated land—New Brunswick to Minnesota and Nebraska, south to Florida, Louisiana, and Texas; also on the Pacific coast from Alaska to California, in tropical America and throughout much of Eurasia.

IOWA DISTRIBUTION: map 4-d. UNCONFIRMED COUNTY REPORTS: Floyd (107), Mahaska (96), Montgomery (120), Polk (56), Ringgold (120), Winnebago (98, 142), and Worth (142). Also, on maps published by *Pammel* (143) and *Pammel and King* (147), the following additional counties (from which I have not yet seen specimens) are indicated: Audubon, Carroll, Cedar, Crawford, Dickinson, Dubuque, Harrison, Ida, Iowa, Jackson, Jones, Kossuth, Monona, Pocahontas, Pottawatamie, Shelby, Sioux, and Woodbury.

4. *Cyperus schweinitzii* Torr. Ann. Lyc. N. Y. 3: 276. 1836.*C. bushii* Britt. Man. 1044 (in part; not as to type). 1901.<sup>10</sup>

Schweinitz's cyperus—in wet or dry sandy soil or railroad ballast—western New York and eastern Ontario to Minnesota, Saskatchewan, and Washington, south to Indiana, Missouri, Texas, Colorado, and Oregon.

<sup>10</sup> See also the notes under *Cyperus filiculmis*, species No. 7.

IOWA DISTRIBUTION: map 4-e. UNCONFIRMED COUNTY REPORTS: Harrison (156, 159), Jasper (107), Linn (133), Mahaska (96), and Scott (99, 107).

5. *Cyperus strigosus* L. Sp. Pl. 47. 1753.

*C. strigosus* var. *robustior* Britt. Bul. Torrey Club 13:221. 1886.

*C. strigosus* var. *capitatus* Britt. l. c.

*C. strigosus* var. *compositus* Britt. l. c.

Straw-colored cyperus—this species may be found in almost any sort of habitat except dense woodland or forest—Maine to Ontario, Saskatchewan, and Washington, south to Florida, Louisiana, Texas, and California.

IOWA DISTRIBUTION: map 4-f. UNCONFIRMED COUNTY REPORTS: Floyd (107), Hardin (148), Harrison (107), and Worth (142). NOTES: (a) One report of the var. *capitatus* for Emmet county (107) was based upon a misidentification of a specimen of *C. engelmanni*. (b) Because of the great diversity of habitats in which this species grows, it assumes many different growth forms. Some of these have been described as varieties, but when a large series of specimens is studied it is impossible to clearly separate these "varieties." For this reason they are not recognized here as being distinct from the species. Fernald (36) has discussed the correct authorship of the varieties included in synonymy above, and also the case for recognition of some of the varieties. O'Neill (60) has recently summarized the evidence against the recognition of varieties under this species.

6. *Cyperus erythrorhizos* Muhl. Gram. 20. 1817.

*C. erythrorhizos* var. *pumilus* Engelm. ex Britt. Bul. Torrey Club 11: 85. 1884.

Red-rooted cyperus—a common species of alluvial pond and stream banks; also found in other wet and marshy places—Massachusetts and southern Ontario to Minnesota, Nebraska, and Washington, south to Florida, Texas, and California.

IOWA DISTRIBUTION: map 4-g. UNCONFIRMED COUNTY REPORTS: Decatur (87), Hardin (141), Muscatine (99, 107, 138), Polk (56), and Story (107, 124, 132). NOTE: The var. *pumilus* is merely a depauperate phase of the species and is frequently found around or in dried-out ponds and ditches; it certainly does not merit varietal recognition.

7. *Cyperus filiculmis* Vahl, Enum. 2:328. 1806.

*C. bushii* Britt. Man. 1044 (as to type). 1901.

*C. filiculmis* var. *macilentus* Fern. Rhodora 8:128. 1906.

Slender cyperus—usually in open dry fields or pariries, frequently in sand or sandy soil—New Hampshire to Ontario, Minnesota, and Nebraska, south to Florida, Texas, and northern Mexico.

IOWA DISTRIBUTION: map 4-h. UNCONFIRMED COUNTY REPORTS: Clinton (107), Floyd (107), Hancock (107), Linn (107), Mahaska (96), and Story (107, 132). NOTES: (a) Reports of this species from Louisa County (107) and from Wright County (107) are based on mis-

identifications of specimens of *C. schweinitzii*. (b) Because of the possibility of confusion between robust forms of this species and forms of *C. schweinitzii* with smooth culms and condensed spikelet clusters, some of the unconfirmed reports listed above may refer to that species. (c) O'Neill (60) has shown that *C. bushii*, as recognized by Britton, consisted of a mixture of this species and *C. schweinitzii*, and that the type is clearly referable here. The var. *macilentus* does not seem to merit the status of either a variety or a form.

8. *Cyperus diandrus* Torr. Cat. Pl. N. Y. 90. 1819.

Low cyperus—a species of low moist ground or marshy places, occasionally found on river banks—New Brunswick to Minnesota and North Dakota, south to South Carolina, Tennessee, Kansas, and Colorado.

IOWA DISTRIBUTION: map 4-i. SPECIMENS EXAMINED: Clinton County, Lyons, September 4, 1896, *Pammel* (G, ISC, NY, SUI). Emmet County, Estherville, August 18, 1922, *Wolden* 655 (ISC, W). Muscatine County, Moscow, without date, *Barnes and Miller* (ISC). Palo Alto County, Booth Township, August 14, 1943, *Hayden* 3173 (ISC); Freedom Township, Medium Lake, September 22, 1940, *Hayden* 8334 (ISC). NOTES: (a) The following county reports of this species are based on misidentifications of *C. rivularis* and should be referred to that species: Black Hawk (104), Dickinson (136, 159), Emmet (142), Fayette (117, 126), Johnson (164), Muscatine (138), Poweshiek (107), Story (107, 132, 139), Winnebago (142), Winneshiek (107, 126, 142, 155), and Worth (142). (b) The following reports, apparently unsubstantiated by specimens, may refer to either this species or to *C. rivularis*: Cerro Gordo (142), Dubuque (145), Fremont (107), Hamilton (142), Johnson (119, 141), Linn (133), Lyon (153), Scott (99), Winnebago (98), and Wright (142).

9. *Cyperus rivularis* Kunth, Enum. 2:6. 1837.

*C. diandrus* var. *castaneus* Torr. Ann. Lyc. N. Y. 3:352. 1836.

Shining cyperus—a species of low wet ground, especially, along streams, ponds, sloughs, and swamps—Maine to southern Ontario and Minnesota, south to North Carolina, Tennessee, Missouri, and Kansas; also in South America.

IOWA DISTRIBUTION: map 4-j. UNCONFIRMED COUNTY REPORTS: Floyd (107), Hardin (148), Harrison (107), and Scott (99, 107). NOTE: see also the comments under *C. diandrus*, above.

10. *Cyperus odoratus* L., var. *squarrosus* (Britt.) Gilly, comb. nov.

"*C. ferax*" of some American authors, in part; not of L. C. Rich. 1792 (BB2, G).

"*C. speciosus*" of some American authors, in part; not of Vahl 1806 (B, BB, BB2, R).

"*C. michauxianus*" of some Iowa authors; not of Schultes 1824.

*C. ferruginescens* Böckl. Linnaea 36:396. 1870.

*C. speciosus* var. *squarrosus* Britt. Bul. Torrey Club 13:214. 1886.

*C. speciosus* var. *ferruginescens* (Böckl.) Britt. Mem. Torrey Club 5:61. 1894 (BB).

*C. ferax* ssp. *speciosus* var. *squarrosus* (Britt.) Kük. Pflanzenr. 101[IV. 20]:620. 1936.

Coarse cyperus—a species of wet or sandy soil—species distribution: Massachusetts to Ontario and Minnesota, south to Florida and Texas; also in California; rather widespread through tropical America; the var. *squarrosus* is essentially restricted to the Mississippi Valley, the Great Lakes area, and the Western Gulf Coastal Plain.

IOWA DISTRIBUTION: map 4-k. UNCONFIRMED COUNTY REPORTS: Chickasaw (126), Clayton (126), Harrison (107), Lee (121), Ringgold (119), and Van Buren (107). NOTES: (a) The correct name to use for this entity has long been in dispute. Fernald (36) considered the Mississippi Valley plants to be distinct from those of the southeastern coastal plain and of the tropics (to which he applied the epithet *ferax*), and decided that *C. ferruginescens* was the proper appellation for our plants of the interior. More recently, however, O'Neill (60, and personal communication), Dandy (*in* Exell: 22) and Fernald (34) have quite conclusively demonstrated that *C. odoratus* must be used for the tropical and southeastern United States plants of this complex; synonymy of the species includes *C. ferax* L. C. Rich. Act. Soc. Hist. Nat. Paris 1: 106. 1792; *C. speciosus* Vahl, Enum. 2:364. 1806; and *C. michauxianus* Schultes, Mant. 2:123. 1824. (b) I am unable, however, to satisfactorily separate the interior and Western Gulf Coastal Plain plants from those of the rest of the species range. It seems to me that this interior and western phase of the species, with the thinner, dull-brown to greenish scales, is better treated as a variety.

# 11. *Cyperus engelmanni* Steud. Syn. Pl. Cyp. 47. 1855.

Engelmann's cyperus—moist sandy swamps or prairie sloughs, or along pond or lake shores; sometimes in dried-up mud, marl, or other calcareous soil—Massachusetts to southern Ontario, Wisconsin, and Iowa, south to New Jersey, Indiana, and Missouri.

IOWA DISTRIBUTION: map 4-l. NOTE: A report of this species from Van Buren County (123) was based on a misidentification of a specimen of *C. strigosus*.

## SPECIES OF CYPERUS DOUBTFULLY OR ERRONEOUSLY REPORTED FOR IOWA

*Cyperus flavescens* L.—Although I have not seen the specimen on which the report for Hardin County (148) is based, I believe it probably is *C. rivularis*. *C. flavescens*, a species of the coastal plain from Maine to Florida and Mexico, and which extends into the lower Mississippi embayment area, is known in the interior only around the Great Lakes.

*Cyperus refractus* Engelm.—The report of this species for Van Buren County (123) was based on a misidentification of a specimen of *C. esculentus*; the report from Linn County (133) has not been confirmed.

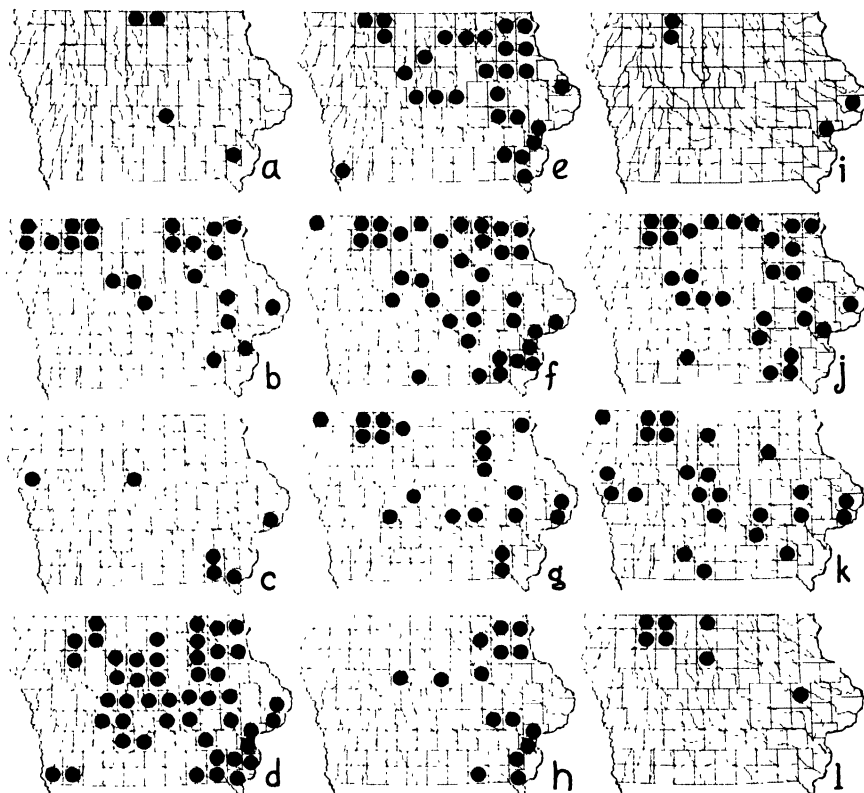


FIGURE 4. Distribution Maps of Iowa Cyperaceae.

- a—*Dulichium arundinaceum*.
- b—*Cyperus aristatus*.
- c—*Cyperus acuminatus*.
- d—*Cyperus esculentus*.
- e—*Cyperus schweinitzii*.
- f—*Cyperus strigosus*.
- g—*Cyperus erythrorhizos*.
- h—*Cyperus filiculmis*.
- i—*Cyperus diandrus*.
- j—*Cyperus rivularis*.
- k—*Cyperus odoratus* var. *squarrosus*.
- l—*Cyperus engelmanni*.



## A SPECIES OF CYPERUS TO BE EXPECTED IN IOWA

*Cyperus tenuifolius* (Steud.) Dandy<sup>11</sup>—This species almost certainly grows in southeastern Iowa, although I have seen no specimens as yet; it is to be expected in sandy or alluvial soil along ponds, streams, or rivers. It has been collected in adjacent Illinois and Missouri.

## 3. HEMICARPHA Nees &amp; Arn. Edinb. New Phil. Jour. 17:263. 1834.

A small genus, of about five or six species, with the center of distribution in the tropics. Only one species is known in Iowa.

1. *Hemicarpha micrantha* (Vahl) Britt. Bul. Torrey Club 15:104. 1888.  
*Scirpus micranthus* Vahl, Enum. 2:254. 1806.  
*S. subsquarrosus* Muhl. Gram. 39. 1817.  
*H. subsquarrosa* (Muhl.) Nees in Mart. Fl. Bras. 2(1):61. 1842.  
*H. drummondii* Nees in Mart. l. c., 62.  
*H. micrantha* var. *drummondii* (Nees) Friedland, Amer. Jour. Bot. 28:860. 1941.  
*H. micrantha* var. *minor* (Schrad.) Friedland, l. c.

Small hemicarpha—usually found along stream or pond banks, sometimes in sandy soil; apparently never long-persistent in any locality toward the northern limits of its range—Maine to Michigan, Minnesota, Nebraska, and Arizona, south to Florida, Louisiana, Texas, and Mexico; also in Washington and California, and widespread in tropical America.

IOWA DISTRIBUTION: map 5-a. SPECIMENS EXAMINED: Black Hawk County, Waterloo, August, 1899, *Hitchcock* (M), September 1, 1893, *Newton* (T). Johnson County, 1899, *Fitzpatrick and Fitzpatrick* (ISC), September, 1894, *Shimek* (M, F, SUI). Story County, near Story City, July 17, 1891, *Pammel* (ISC), August 28, 1934, *Fults* 2816 (ISC). Webster County, Fort Dodge, *Paige* (ISC), August 8, 1906, *Oleson* (ISC). UNCONFIRMED COUNTY REPORTS: Hardin (148), Muscatine (99, 107), and Scott (99, 107). NOTES: Friedland (37) has recently monographed the American species of this genus; he reports two out of the three varieties, which he recognizes, for Iowa. Thus far, however, I have seen only specimens of var. *minor*. If one so desires, the two varieties may be distinguished, on the basis of the structure of the perianth scale,<sup>12</sup> as follows:

<sup>11</sup> *C. tenuifolius* (Steud.) Dandy in Exell, Cat. Vasc. Pl. S. Tomé 363. 1944; *Kyllinga pumila* Michx. Fl. Bor.-Am. 1:28. 1803, not *Cyperus pumilus* L. 1756; *Kyllinga tenuifolia* Steud. Syn. Pl. Cyp. 69. 1855; *C. densicaespitosus* Mattf. and Kük. Pflanzenr. 101[IV. 20]:597. 1936. This species is usually included in the segregate genus *Kyllinga* by most authors. *Kyllinga*, however, is an artificial grouping of species of *Cyperus* in which the spikelets have been reduced to a 1-flowered condition. There seems to be no clear distinction, other than the reduction in flower number per spikelet, between this group and typical *Cyperus*; since the species commonly placed in *Kyllinga* are derived from the several diverse sections of the genus *Cyperus*, there seems to be no justifiable reason for maintaining *Kyllinga* as a genus.

<sup>12</sup> Blaser (11) has recently interpreted this perianth scale as a prophyll (i.e., the lowermost leaf on the "branch" terminated by the flower); additional anatomical work should be carried out to determine which interpretation is correct. If the scale is shown to be a prophyll, then *Hemicarpha* should be placed in the *Lipocarpha* alliance. *Hemicarpha*, and also *Fimbristylis*, seem scarcely separable from the genus *Scirpus*.

- (a) perianth scale as long as or longer than the achene, at most only slightly erose, and with 3 to 5 vascular strands.....the var. *drummondii*
- (b) perianth scale shorter than achene, deeply bifid, variously cut or lobed, or sometimes absent; vascular strands short and inconspicuous or lacking .....the var. *minor*

## 4. SCIRPUS L. Sp. Pl. 47. 1753.

A large genus with nearly world-wide distribution but most common in the temperate zones; more than 250 species are regarded as distinct. Ten species have been found in Iowa.

## KEY TO THE IOWA SPECIES

- A. Involucre of a single, scarcely leaf-like, bract; inflorescence appearing to be lateral on the culm.
  - B. Culms triangular; spikelets few, sessile or almost so in a capitate cluster.
    - C. Scales merely acute or mucronate at apices; perianth bristles longer than the achene; styles 3, the achene unequally triangular..... 1. *S. torreyi*
    - CC. Scales bifid at apices, the midrib prolonged between the teeth as a prominent mucro; perianth bristles shorter than the achene; styles 2, the achene lenticular (or, sometimes, styles 3 and the achene obtusely triangular) ..... 2. *S. americanus*
  - BB. Culms terete; spikelets numerous, in small clusters of 1 to 7, arranged in compound umbels or umbel-like corymbs.
    - C. Stigmas 3; achenes triangular, the terminal apiculation one-fourth to one-third as long as the achene body; scales essentially glabrous, with erose but not ciliate margins ..... 3. *S. heterochaetus*
    - CC. Stigmas 2; achenes lenticular, the terminal apiculation but one-eighth as long as the achene body; scales more or less pubescent, the margins erose or entire and usually conspicuously ciliate.
      - D. Achenes 2 mm. or less in length; scales broadly ovate, scarcely (if at all) longer than the achenes; spikelets ovoid, less than 1 cm. long (except in the forma *megastachyus*) ..... 4. *S. validus*  
var. *creber*
      - DD. Achenes 2.5 mm.-3 mm. long; scales lanceolate-oblong, about one-third longer than the achenes; spikelets oblong-cylindrical, normally 1 cm.-2 cm. long ..... 5. *S. acutus*
- AA. Involucre of several flat, leaf-like bracts; inflorescence appearing to be terminal.
  - B. Spikelets large, 1 cm.-3 cm. long; achenes 3 mm.-4 mm. long.
    - C. Stigmas 2; achenes lenticular; spikelets capitate or umbellate, on short pedicels ..... 6. *S. paludosus*
    - CC. Stigmas 3; achenes triangular; spikelets long-pedicelled in a loose open inflorescence ..... 7. *S. fluvialis*
  - BB. Spikelets small, 0.5 mm.-1 mm. long; achenes 1.5 mm. long or less.
    - C. Perianth bristles downwardly barbed, scarcely as long as the achenes, or sometimes absent; culms usually solitary; plants with thick, scaly rhizomes.
      - D. Scales acute, acuminate or mucronate, greenish-brown to dark brown or blackish, 1.5 mm.-2 mm. long, only slightly longer than the achenes; leaves medium- to dark-green ..... 8. *S. atrovirens*
      - DD. Scales with midribs prolonged as more or less scabrous awns, one-fourth as long to almost as long as the scale bodies, the scales usually straw-colored or light-brown, 2 mm.-3 mm. long, about twice as long as the achenes; leaves pale-green ..... 9a. *S. atrovirens*  
var. *pallidus*
    - CC. Perianth bristles smooth, barbless, flexuous, considerably longer than the achenes; culms tufted or cespitosely clustered; plants non-rhizomatous.
      - D. Spikelets cylindrical, smooth, shining, 5 mm.-10 mm. long; perianth bristles not exceeding scales at maturity..... 9. *S. lineatus*
      - DD. Spikelets ovoid, dull, appearing wooly at maturity, about 5 mm. or less in length; perianth bristles much exceeding the scales at maturity.

- E. Spikelets in glomerules of 3 or more, all sessile or nearly so; involuclers reddish-brown or dull gray-brown with blackish bases ..... 10. *S. cyperinus*
- EE. Spikelets mostly pedicelled (at least the lateral ones in each cluster are); involuclers various in color.
- F. Involuclers stramineous, brown or red-brown (terra-cotta); perianth hairs brownish or reddish.
- G. Involuclers dull-brown or stramineous; spikelets dull-brown, drab or straw-colored; perianth hairs brownish ..... 10a. *S. cyperinus* var. *laxus*
- GG. Involuclers bright reddish-brown; spikelets reddish-brown; perianth hairs reddish ..... 10b. *S. cyperinus* var. *rubricosus*
- FF. Involuclers black or blackish; spikelets dull-brown to blackish; perianth hairs whitish or pale ..... 10c. *S. cyperinus* var. *brachypodus*

1. *Scirpus torreyi* Olney, Proc. Prov. Frank. Soc. 1:32. 1847.

Torrey's bulrush—in swamps or occasionally along stream or lake margins—Maine to Manitoba, south to Pennsylvania, Indiana, Missouri, and North Dakota.

IOWA DISTRIBUTION: map 5-b. SPECIMEN EXAMINED: Clinton County, July 10, 1878, *Butler* 3 (G). NOTE: This species was previously unreported for the state except as included in the range given for the species in "Gray's" *Manual* (66) and *Rydberg's Flora* (67).

2. *Scirpus americanus* Pers. Syn. 1:68. 1805.

*S. pungens* Vahl, Enum. 2:255. 1806.

*S. pungens* var. *polyphyllus* Böckl. Linnaea 36:709. 1870.

*S. americanus* var. *polyphyllus* (Böckl.) Beetle, Amer. Jour. Bot. 30:399. 1943.

American bulrush—marshes, bogs, low swampy ground, lake and pond shores, and prairie sloughs are the usual habitat for this species—Newfoundland to Manitoba and British Columbia, south to Florida, Louisiana, Texas, and California; also in Chile.

IOWA DISTRIBUTION: map 5-c. UNCONFIRMED COUNTY REPORTS: Harrison (159), Muscatine (99, 107), Scott (99, 107), and Winneshiek (142, 155). NOTE: Beetle (6) has recently appended a number of varieties to this species. One of these varieties is found in Iowa, along with the typical phase of the species, and may be separated, if one wishes to do so, as follows:

- (a) style 2-branched; 2, or rarely 3, of the basal sheaths bearing blades ..... typical *americanus*
- (b) style 3-branched; 3 or more of the basal sheaths terminating in leafy blades ..... the var. *polyphyllus*

3. *Scirpus heterochaetus* Chase, Rhodora 6:70. 1904.

Pale great bulrush—in swamps, sloughs, and along lake shores, in 1–3 feet of water—Quebec and Massachusetts to North Dakota, south to Illinois, Missouri, and Oklahoma; also in Idaho, Washington, and Oregon.

IOWA DISTRIBUTION: map 5-d. NOTE: see discussion under *S. validus*, var. *creber*, below.

4. *Scirpus validus* Vahl, var. *creber* Fern. Rhodora 45:283. 1943.

"*S. lacustris*" of many American authors; not of L. 1753 (B, BB).

"*S. validus*" of many American authors; not of Vahl 1806 (BB2, G, R).

*S. validus* var. *creber* f. *megastachyus* Fern. Rhodora 45:283. 1943.

Great bulrush—common in sloughs, ditches, swamps, and in water around margins of ponds and lakes—Newfoundland to Manitoba and British Columbia, south to Georgia, Tennessee, Missouri, Oklahoma, Texas, New Mexico, northern Mexico, and California; reported from the Hawaiian Islands, from Manchuria and Japan, and south to Australia and New Zealand.

IOWA DISTRIBUTION: map 5-e. UNCONFIRMED COUNTY REPORTS: Appanoose (119), Butler (107), Cerro Gordo (141, 142), Clayton (118), Dubuque (118), Floyd (107), Fremont (119), Hamilton (137), Hardin (148), Harrison (159), Lee (121), Mahaska (96), Muscatine (99, 107, 138), Scott (99, 107, 134), Shelby (119), Woodbury (136), Worth (142), and Wright (142). NOTES: (a) Some of the earlier of these reports, particularly those listed as "*S. lacustris*," may refer to either *S. acutus* or *S. heterochaetus* instead of to this species. (b) The Decatur County record on the map is based on specimens examined by A. A. Beetle. (c) Reports of this species from Cerro Gordo County (107), Fremont County (88), and Pottawattamie County (136) were based on misidentifications of specimens of *S. acutus*, and a report from Hancock County (107) was based on a misidentification of a specimen of *S. heterochaetus*. (d) Fernald (33) has recently shown that almost all of North American material is to be included in the var. *creber*; the true *C. validus* Vahl (Enum. 2: 268. 1806) is confined to tropical America and Florida. The forma *megastachyus* has larger spikelets (9 mm. to 15 mm. long) and larger achenes (2.3 mm. to 2.8 mm. long and 1.4 mm. to 1.8 mm. broad).

5. *Scirpus acutus* Muhl. ex Bigelow, Fl. Bost. 15. 1814.

*S. occidentalis* (Wats.) Chase, Rhodora 6:68. 1904 (BB2, G).

Viscid great bulrush—like the two preceding species, this is found along the edges of lakes and streams and in swamps—Newfoundland to British Columbia, south to North Carolina, Tennessee, Oklahoma, New Mexico, Arizona, and California.

IOWA DISTRIBUTION: map 5-f. NOTE: A report of this species from Black Hawk County (104) was based on a misidentification of a specimen of *S. heterochaetus*; see also the discussion under *S. validus*, above.

6. *Scirpus paludosus* A. Nels. Bul. Torrey Club 26:5. 1899.

*S. campestris* Britt. in Britt & Brown, Ill. Fl. 1:267. 1896; not of Roth 1795 (B, BB, BB2).

*S. campestris* var. *paludosis* (A. Nels.) Fern. Rhodora 2:241. 1900 (G).

Prairie bulrush—swamps, marshes, and prairie sloughs, in fresh or saline water—Manitoba and Minnesota to British Columbia, south to Iowa, Kansas, Arizona, and northern Mexico; local in the east from New

Brunswick to Quebec and south to New Jersey; also in South America and reported from the Hawaiian Islands.

IOWA DISTRIBUTION: map 5-b. SPECIMENS EXAMINED: Palo Alto County, Booth Township, Rush Lake, July 23, 1942, *Hayden* 7604 (ISC), August 14, 1943, *Hayden* 3163 (ISC). NOTE: The eastern material has recently been segregated by Fernald (33) as var. *atlanticus*.

7. *Scirpus fluviatilis* (Torr.) A. Gray, Man. 527. 1848.

"*S. maritimus*" of some authors; not of L. 1753.

*S. maritimus* var. *fluviatilis* Torr. Ann. Lyc. N. Y. 3: 324. 1836.

River bulrush—in marshes, swamps, or in deep or shallow water along streams and lakes—southern Massachusetts and Quebec to Minnesota, North Dakota, Montana, and Washington, south to northern Virginia, northern Indiana, Missouri, Kansas, New Mexico, and California.

IOWA DISTRIBUTION: map 5-g. UNCONFIRMED COUNTY REPORTS: Hamilton (142), Hancock (107, 124), Hardin (148), Johnson (124, 164), Kossuth (107), Mahaska (97), Story (92, 107, 124, 132), and Worth (142). NOTES: (a) The species has also been reported, in letter, by M. McDonald for Jefferson County (Lockridge Township) and Louisa County (in the Conesville marshes); no specimens were collected at either locality. (b) The Decatur County record on the map is based on specimens collected by Fitzpatrick and by J. P. Anderson which have been examined by A. A. Beetle.

8. *Scirpus atrovirens* Willd. Enum. Pl. Hort. Berol. 79. 1809.<sup>13</sup>

*S. georgianus* Harper, Bul. Torrey Club 27: 331. 1900.

*S. atrovirens* var. *georgianus* (Harper) Fern. Rhodora 23: 134. 1921.

Dark-green bulrush—found in swamps, sloughs, prairie swales, and along pond and stream banks—Nova Scotia to Manitoba, and Saskatchewan, south to Georgia, Louisiana, and Texas.

IOWA DISTRIBUTION: map 5-h. UNCONFIRMED COUNTY REPORTS: Benton (107), Dubuque (118, 145), Floyd (107, 124), Hardin (148), Harrison (107), Mahaska (96), Montgomery (120), Polk (124), Scott (99, 107), Woodbury (136), and Worth (142). NOTES: (a) The Shelby County record on the map is based on a Fitzpatrick specimen examined by A. A. Beetle. (b) This polymorphic species has been variously handled by different authors; some considering it to be composed of several varieties, and others considering these varieties as distinct species. Some few specimens can be clearly referred to *georgianus* and numerous others to *pallidus*, but the intergradations between these extremes and typical *S. atrovirens* are so numerous and so varied that I cannot accept specific status for either of the extremes, nor even varietal status for *georgianus*. Specimens which show evident combinations of characters and intergradations between the two extremes are not rare. There appears to be no clear geographical segregation of the following variety,

<sup>13</sup> Usually attributed to Muhlenberg (Gram. 43. 1817), but Beetle (8) has shown that the real author of the species is Willdenow.

although most specimens of it come from the western part of the species range; the *georgianus* phase is more plentiful in the southeast.

8a. *Scirpus atrovirens* Willd., var. *pallidus* Britt. Trans. N. Y. Acad. Sci. 9: 14. 1889.

*S. pallidus* (Britt.) Fern. Rhodora 8: 162. 1906.

Pale bulrush—swamps, sloughs, and prairies swales, sometimes along pond and stream banks—Minnesota to Wyoming, south to Texas and New Mexico; also in the Great Basin.

IOWA DISTRIBUTION: map 5-i. NOTE: See discussion, above, under the species.

9. *Scirpus lineatus* Michx. Fl. Bor.-Amer. 1: 32. 1803.

*Eriophorum lineatum* (Michx.) Benth. & Hook. Gen. Pl. 3: 1052. 1883.

Reddish bulrush—in swamps or boggy prairie areas, or along ponds and streams—Maine, New Hampshire, and Ontario to Manitoba and Oregon, south to northern Florida, Alabama, and Texas.

IOWA DISTRIBUTION: map 5-j. UNCONFIRMED COUNTY REPORTS: Hamilton (142), Muscatine (99, 107), Scott (99, 107), and Winnebago (98). NOTE: The records on the map for Johnson and Appanoose Counties are based on data sent in letter by A. A. Beetle, who examined the specimens.

10. *Scirpus cyperinus* (L.) Kunth, Enum. 2: 170. 1837.<sup>14</sup>

*Eriophorum cyperinum* L. Sp. Pl. ed. 2. 77. 1762.

*S. thyrsoiflorus* Willd. Enum. Pl. Hort. Berol. 78, *nomen superfluum*. 1809.

*S. eriophorum* var. *cyperinus* (L.) A. Gray, Man. ed. 2. 501. 1856.

*S. eriophorum* var. *andrewsii* Fern. Proc. Amer. Acad. 34: 501. 1899.

*S. eriophorum* var. *condensatus* Fern. l. c.

*S. cyperinus* var. *andrewsii* (Fern.) Fern. Rhodora 2: 16. 1900.

*S. cyperinus* var. *condensatus* (Fern.) Fern. l. c.

*S. cyperinus* var. *pelius* Fern. Rhodora 8: 164. 1906.

Wool-grass—wet meadows, prairie sloughs, swamps, boggy spots, and along sluggish streams—Newfoundland to Ontario, Manitoba, and Saskatchewan, south to Virginia, Tennessee, and Arkansas.

IOWA DISTRIBUTION: map 5-k. NOTES: (a) The several "species" included in synonymy under this species and the three varieties which I am here recognizing do not appear to merit independent recognition. Achenes of all are essentially the same in size, proportions, color, and surface markings. There is some variation in size and shape of the spikelet scales, but the variation (obtuse, acute, or mucronate apices; smooth or scabrid mucro; entire or erose margins) may frequently be assorted in different manners on individual plants. Degree of pedicellation of spikelets, and

<sup>14</sup>Only specific and varietal names have been included in the synonymy of this species and the three varieties which I am recognizing. Numerous forms have been proposed by several authors, but it seems needless to mention them here.

the color of involucels and bases of the involucre bracts have been used as the strongest characters for the separation of the "species." It is of more than passing interest to note that most of the varieties usually recognized under these "species" differ from the species to which they are appended in having involucels and involucre bracts of the color characteristic of another of the "species." The degree of pedicellation, or its lack, and the resultant diffuseness of inflorescence or agglomeration of spikelets and spikelet clusters does not seem to be correlated with the color differences. I am forced to the conclusion, after study of a large series of specimens, that this species complex is best treated as a single polymorphic species composed of a number of more or less geographically segregated varieties which are characterized by reasonably constant characters of color and inflorescence structure.<sup>15</sup>

10a. *Scirpus cyperinus* (L.) Kunth, var. *laxus* (A. Gray) Wats. & Coult. in A. Gray, Man. ed. 6. 582. 1890.

*S. eriophorum* var. *laxus* A. Gray, Man. ed. 2. 501. 1856.

*S. pedicellatus* Fern. Rhodora 2:16. 1900.

*S. pedicellatus* var. *pullus* Fern. l. c., 17.

*S. atrocinctus* var. *grandis* Fern. l. c.

Long-stalked wool-grass—habitat essentially as in the species—Connecticut and eastern Quebec to Minnesota, south to New York, Ohio, Indiana, and Iowa.

IOWA DISTRIBUTION: map 5-l. SPECIMENS EXAMINED: Muscatine County, Muscatine, summer of 1935, *Estle and Brown* (ISC, NY). Winneshiek County, Calmar Township, June 30, 1933, *Tolstead* (ISC). NOTE: The record on the map for Floyd County is based on a specimen examined by A. A. Beetle.

10b. *Scirpus cyperinus* (L.) Kunth, var. *rubricosus* (Fern.) Gilly, comb. et stat. nov.

*S. eriophorum* Michx. Fl. Bor.-Amer. 1:33, *nomen superfluum*. 1803.

*S. cyperinus* var. *eriophorum* (Michx.) Kuntze, Rev. Gen. Pl. 2: 757. 1891.

*S. rubricosus* Fern. Rhodora 47:124. 1945.

Southern wool-grass—habitat essentially as in the species—along coastal plain from Massachusetts to Florida and west to eastern Texas, in scattered localities west of the Appalachian Mountains, and north in the Mississippi Valley to southern Iowa.

IOWA DISTRIBUTION: map 5-l. SPECIMENS EXAMINED: Davis County, Lick Creek Township, June 26, 1939, *Hayden* 9196 (ISC), September 7, 1940, *Hayden* 8330 (ISC).

<sup>15</sup> Fernald (34) has shown that both Michaux and Willdenow, the former in describing his *S. eriophorum* (see below, the var. *rubricosus*), and the latter in publishing his *S. thyrsiflorus*, not only failed to take up the earlier Linnaean epithet *cyperinus* when publishing their new species, but also included it in direct synonymy. The epithets, then, which they proposed are illegitimate since they were superfluous when published; see Art. 60 of the *International Rules of Botanical Nomenclature* (12).

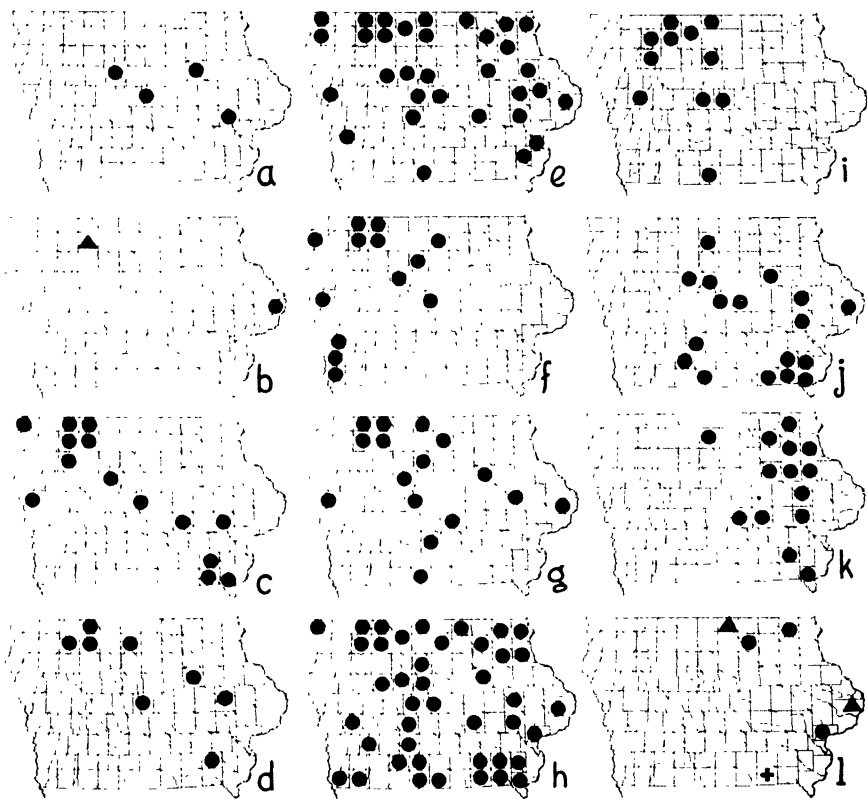


FIGURE 5. Distribution Maps of Iowa Cyperaceae.

- a—*Hemicarpha micrantha*.
- b—*Scirpus torreyi* (●) and *Scirpus paludosus* (▲).
- c—*Scirpus americanus*.
- d—*Scirpus heterochaetus*.
- e—*Scirpus validus* var. *creber*.
- f—*Scirpus acutus*.
- g—*Scirpus fluviatilis*.
- h—*Scirpus atrovirens*.
- i—*Scirpus atrovirens* var. *pallidus*.
- j—*Scirpus lineatus*.
- k—*Scirpus cyperinus*.
- l—*Scirpus cyperinus* var. *laxus* (●), *Scirpus cyperinus* var. *rubricosus* (+), and *Scirpus cyperinus* var. *brachypodus* (▲).



10c. *Scirpus cyperinus* (L.) Kunth, var. **brachypodus** (Fern.) Gilly, comb. nov.

*S. atrocinctus* Fern. Proc. Amer. Acad. 34: 502. 1899.

*S. atrocinctus* var. *brachypodus* Fern. l.c., 503.

*Eriophorum cyperinum* var. *brachypodum* (Fern.) Farw. Ann. Rpt. Mich. Acad. Sci. 6: 204. 1904.

Northern wool-grass—much the same habitat as the species, with the inclusion of bogs and cold springy spots—Newfoundland to James Bay and British Columbia, south to New Jersey, Pennsylvania, Michigan, Iowa, Manitoba, and Saskatchewan.

IOWA DISTRIBUTION: map 5-l. SPECIMENS EXAMINED: Clinton County, July 1, 1878, Butler 7 (G). Worth County, bog near Fertile, September, 1908, Pammel (ISC).

#### SPECIES OF SCIRPUS DOUBTFULLY OR ERRONEOUSLY REPORTED FOR IOWA

*Scirpus hallii* A. Gray—This species has been reported from Muscatine County (99, 107); I have not been able to find the specimen on which these reports are based.

*Scirpus subterminalis* Torr.—This species has been reported from Jefferson County (123) but the specimen which formed the basis of that report is *S. americanus* Pers.

#### 5. ERIOPHORUM L. Sp. Pl. 52. 1753.

A small genus of boreal, alpine, and north-temperate zone species, characterized by the elongated and compound perianth bristles from whence the common name, "cotton-grass," is derived; otherwise, much as in *Scirpus*. About ten or twelve species, two of which have been found in Iowa, are recognized as distinct.

#### KEY TO THE IOWA SPECIES

- A. Blade of the uppermost leaf as long as or longer than the sheath; leaves 3 mm.-8 mm. wide; sheaths blackish-margined at mouth; scales with conspicuous hyaline tips, the midribs not reaching the apices..... 1. *E. angustifolium*
- AA. Blade of the uppermost leaf much shorter than its sheath; leaves 2 mm. or less in width; scales not hyaline-tipped, the midrib prominent to the apices ..... 2. *E. gracile*

1. *Eriophorum angustifolium* Roth, Tent. 1: 24. 1788.

"*E. polystachyon*" of many authors; not of L. 1753 (BB).

*E. angustifolium* var. *majus* Schultz, Fl. Starg. Suppl. 5. 1819.

Tall cotton-grass—in bogs, hanging bogs, wet prairie sloughs, or marshes—throughout much of arctic North America, south to Maine, Ontario, Illinois, Iowa, South Dakota, Montana, and Oregon.

IOWA DISTRIBUTION: map 6-b. UNCONFIRMED COUNTY REPORTS: Hamilton (142), Hardin (148), Mitchell (167), Muscatine (99), Scott (99), and Story (100, 130).

2. *Eriophorum gracile* Koch, Cat. 2:259. 1800.

"*E. gracile* var. *paucinervium*" of some Iowa authors; not of Engelm. 1852.

Slender cotton-grass—almost wholly confined to bogs—Newfoundland and Hudson Bay to British Columbia, south to New York, New Jersey, Illinois, Nebraska, and California.

IOWA DISTRIBUTION: map 6-a. SPECIMENS EXAMINED: Emmet County, 1878, *Cratty* (ISC), June, 1893, *Cratty* (SUI); Armstrong, June 6, 1883, *Cratty* (ISC), without date, *Paige* (ISC). Webster County, Fort Dodge, May 23, 1905, *Oleson* (ISC). UNCONFIRMED COUNTY REPORTS: Dickinson (158) and Wright (107). NOTE: *E. tenellum* Nutt. (the true *E. gracile* var. *paucinervium* Engelm.) differs from this species by its rough culms and uppermost leaf blades longer than their sheaths; it is known from Illinois and Wisconsin, and thus might be expected in northeastern Iowa.

SPECIES OF *ERIOPHORUM* ERRONEOUSLY REPORTED FOR IOWA

*Eriophorum viridicarinatum* (Engelm.) Fern.—This species was listed for Iowa in "Gray's" *Manual* (66) on the basis of a misidentification of a specimen of *E. angustifolium* (Fayette County, Fayette, May, 1894, *Fink*) now in the Gray Herbarium. It has also been reported from Mahaska County (97); although I have not seen the specimen on which this report is based, I suspect that it, too, has been misidentified.

6. *FIMBRISTYLIS* Vahl, Enum. 2:285. 1806.<sup>16</sup>

About 125 species are usually recognized in this largely tropical genus; only one species is known in Iowa.

1. *Fimbristylis autumnalis* (L.) R. & S. Syst. 2:97. 1817.

*Scirpus autumnalis* L. Mant. 2:180. 1781.

*Scirpus mucronulatus* Michx. Fl. Bor.-Amer. 1:31. 1803.

*F. geminata* Kunth, Enum. Pl. 2:247. 1837 (BB2).

*F. frankii* Steud. Syn. Pl. Cyp. 111. 1855 (B, G).

*F. mucronulata* (Michx.) Blake, *Rhodora* 20:25. 1918.

*F. autumnalis* var. *mucronulata* (Michx.) Fern. *Rhodora* 37:398. 1935.

Slender fimbristylis—sandy or moist soil—Maine and Ontario to Indiana, Iowa, and Kansas, south to Florida, Louisiana, and Texas; widespread in tropical America.

IOWA DISTRIBUTION: map 6-a. SPECIMENS EXAMINED: Muscatine County, Fruitland, without date, *Barnes and Miller* (ISC); Moscow, without date, *Barnes and Miller* (ISC). UNCONFIRMED COUNTY REPORTS: Lee (107) and

<sup>16</sup> This genus probably should be merged with *Scirpus* from which it is but weakly separated (see Key to the Iowa Genera, page 69). The enlarged base of the deciduous style seems to represent a condition transitional between *Scirpus* and *Bulbostylis*.

Scott (99). NOTES: Blake (10) and Fernald (29) have clarified the nomenclatural problems involved with this species. Iowa material includes both phases of the species which may be distinguished if one desires, as follows:

- (a) umbels usually simple; spikelets ovoid; achenes about 0.75 mm.  
long ..... typical *autumnalis*
- (b) umbels usually compound; spikelets narrowly cylindric; achenes about 0.5 mm.  
long ..... the var. *mucronulata*

7. *BULBOSTYLIS* Kunth ex C. B. Clarke in Hook. f. Fl. Br. Ind. 6:651. 1893.<sup>17</sup>

*Stenophyllus* Raf. Neog. 4. 1825.

An essentially tropical genus of at least 50 species; only one is known in Iowa.

1. *Bulbostylis capillaris* (L.) C. B. Clarke, in Hook. f. Fl. Br. Ind. 6:651. 1893.

*Scirpus capillaris* L. Sp. Pl. 49. 1753.

*Stenophyllus capillaris* (L.) Britt. Bul. Torrey Club 2:30. 1894 (B, BB, BB2, G, R).

*B. capillaris* var. *pycnostachys* Fern. Rhodora 19:154. 1917.

*B. capillaris* var. *crebra* Fern. Rhodora 40:395. 1938.

Slender *bulbostylis*—usually found in more or less sandy soil, sometimes in railroad ballast—Maine to Florida, westward to the Pacific Coast; frequently a waif in the northern part of this range; also widely distributed in the tropics.

IOWA DISTRIBUTION: map 6-c. SPECIMENS EXAMINED: Muscatine County, Fruitland, August, 1896, *Barnes and Miller* (ISC, SUI). UNCONFIRMED COUNTY REPORTS: Dickinson (157, 159), Lee (93), and Scott (99, 107). NOTES: Fernald recognizes several varieties of this species, two of which are included in the single Iowa specimen—which consists of about eight plants—available for study. The var. *pycnostachys* is the typical form of the species, and the var. *crebra* differs from it in having the spikelets of the terminal umbel pedicellate and no sessile spikelets at the base of the leaves.

8. *ELEOCHARIS* R. Br. Prod. 1:209. 1810.

This genus, like *Cyperus*, has an almost world-wide range with its center of distribution in the tropics and sub-tropics. About 150 species are recognized as distinct; ten species are known in Iowa.

#### KEY TO THE IOWA SPECIES

- A. Style-base confluent with the achene; the tubercle, although of definitely different texture than the body of the achene, not forming a distinct apical cap ..... 1. *E. pauciflora*  
var. *fernaldii*

<sup>17</sup> Fernald (30) already has adequately discussed the unfortunate conservation of this generic name over the adequately published *Stenophyllus* Raf.

- AA. Style-base forming an obvious cap-like tubercle on the apex of the achene.
- B. Tubercle more or less conical, pyramidal or sometimes bulbously swollen (if laterally compressed, as sometimes in *E. calva* and *E. macrostachya*, then lighter in color than the achene); achenes lenticular, biconvex or triangular.
- C. Achenes triangular or essentially so; style 3-branched.
- D. Surfaces of the whitish achenes conspicuously transversely cellular-reticulated between prominent longitudinal ridges.
- E. Culms coarse, firm, flattened, and 2-edged (the edges sometimes in-rolled so that the culm appears to be terete), 0.5 mm.–1 mm. wide; plants usually more than 15 cm. tall; scales sharply acute at apices, the triangular apical portion conspicuously white-hyaline..... 2. *E. wolffi*
- EE. Culms capillary, weak, angled, or almost terete, less than 0.5 mm. in diameter; plants dwarfed, usually less than 10 cm. tall; scales obtuse at apices, narrowly (if at all) hyaline-margined ..... 3. *E. acicularis*
- DD. Surfaces of the brown and yellow achenes smooth, minutely pitted, or warty.
- E. Achene 1 mm.–1.5 mm. long, golden yellow, the surface minutely pitted (appearing almost smooth under a low-power hand lens); scales narrowly lanceolate, the apices conspicuously white-hyaline, acuminate, usually deeply bifid ..... 4. *E. compressa*
- EE. Achene 0.75 mm.–1 mm. long, olive-brown or dark-brown (rarely somewhat yellowish when young), the surface either verrucose (warty) or so deeply cellular-pitted as to appear papillose or verrucose; scales rounded to acute at apices, scarcely (if at all) hyaline margined, the apices entire.
- F. Plants 3 dm.–9 dm. tall; culms about 1 mm. or more in diameter; scales dark red-brown; spikelets many-flowered ..... 5. *E. tenuis*
- FF. Plants 6 cm. or less in height; culms filiform; scales green to pale brown; spikelets few-flowered ..... 6. *E. coloradoensis*
- CC. Achenes lenticular or biconvex; style 2-branched.
- D. Tubercle minute, depressed-conic, almost saucer-shaped; achenes black, less than 0.5 mm. long..... 7. *E. atropurpurea*
- DD. Tubercle conspicuous, more or less bulbously swollen at base, subterete or somewhat triangular (occasionally somewhat laterally compressed); achenes yellow or brownish, more than 1 mm. long.
- E. Basal empty scales 2 or 3, oblong, contiguous with the culm, the spike appearing as if inserted into the split apex of the culm; fertile scales narrowly lanceolate, acute to acuminate at the apices, the central portion firm in texture, the marginal areas membranous..... 8. *E. macrostachya*
- EE. Basal empty scale 1, broadly orbicular, spatiform, and encircling the entire base of the spike, the spike often appearing to be obliquely offset from the culm; fertile scales oblong to lanceolate, obtuse and rounded at apices, the entire scale membranous in texture ..... 9. *E. calva*
- BB. Tubercle strongly laterally compressed, appearing as a dark brown triangular wafer with the broadest edge attached to the truncated apex of the lenticular to biconvex, obovate, pale or light brownish achene.
- C. Tubercle of the achene prominent, one-third to one-fourth the height of the achene body; perianth bristles usually longer than the achene; scales more or less orbicular ..... 10. *E. obtusa*
- CC. Tubercle of the achene low, generally no more than one-seventh the height of the achene body; perianth bristles usually shorter than the achene (or sometimes absent); scales oblong or lanceolate..... 10a. *E. obtusa*  
var. *engelmanni*

1. *Eleocharis pauciflora* (Lightf.) Link., var. *fernaldii* Sv. Rhodora 36: 380. 1934.

"*Scirpus pauciflorus*" of many American authors; not of Lightf. 1777 (B, BB, BB2, G).

"*E. pauciflora*" of some Iowa authors; not of Link. 1827 (R).

Few-flowered spike-rush—wet soil along edges of ponds and lakes, bogs, prairie sloughs, muddy ditches—Newfoundland to Quebec, Ontario,

and Michigan, south to New Hampshire, Vermont, New York, Pennsylvania, Indiana, Illinois, and Iowa.

IOWA DISTRIBUTION: map 6-c. SPECIMENS EXAMINED: Emmet County, Emmet Township, June 24, 1931, *Wolden* (ISC), July 14, 1931, *Wolden* 1498 (G, ISC, W). NOTE: Svenson (73) recognizes several additional varieties of this species in the western part of North America. The typical phase of the species is confined to Europe and a few areas in northern Asia.

2. *Eleocharis wolfii* (A. Gray) A. Gray in Patterson, Cat. Pl. Ill. 46. 1876.

Wolf's spike-rush—usually found in wet meadows and prairies, or in shallow water along ponds—southern Indiana to northern Iowa and Saskatchewan, south to eastern Tennessee, Alabama, Louisiana, and Colorado; recently reported from one locality on Long Island, New York (74). IOWA DISTRIBUTION: map 6-d. SPECIMENS EXAMINED: Emmet County, July 15, 1882, *Cratty* (G), June 9, 1886, *Cratty* (ISC, NY, SUI), summer of 1938, *Wolden* (W); Armstrong, 1885, *Cratty* (GRC, ISC). UNCONFIRMED COUNTY REPORTS: Dickinson (157), Johnson (107), and Palo Alto (145). NOTE: A report of this species for Black Hawk County (104) was based on a misidentification of a specimen of *E. acicularis*.

3. *Eleocharis acicularis* (L.) R. & S. Syst. 2: 154. 1817.

Needle spike-rush—in wet prairies or along muddy shores, stream banks, and swamp edges—Greenland, southern Labrador, and Newfoundland to northern Manitoba and British Columbia, south to Florida, Alabama, Tennessee, Missouri, Oklahoma, New Mexico, and California; also in Europe from northern Scandinavia to northern Spain and Italy, in temperate-zone Asia, Japan, and the Caucasus.

IOWA DISTRIBUTION: map 6-e. UNCONFIRMED COUNTY REPORTS: Cerro Gordo (142), Floyd (107), Hamilton (137, 142), Hardin (148), Kossuth (107), Muscatine (99, 107), Poweshiek (107), Scott (99, 107), Winneshiek (142), Woodbury (136), and Worth (142).

4. *Eleocharis compressa* Sull. Amer. Jour. Sci. 42: 50. 1842.

? *Scirpus acuminatus* Muhl. Gram. 27, *nomen ambiguum*. 1817.

? *E. acuminata* (Muhl.) Nees, Linnaea 9: 294. 1835 (B, BB, BB2, G, R).

Flat-stemmed spike-rush—wet or damp places—southern Quebec to Saskatchewan, south to Virginia, northwestern Georgia, Missouri, northern Texas, and Colorado.

IOWA DISTRIBUTION: map 6-f. UNCONFIRMED COUNTY REPORTS: Benton (72), Hancock (107), Story (107), and Wright (172). NOTE: According to Svenson (172), *E. acuminata* Nees is based on the incomplete and unidentifiable description of *Scirpus acuminatus* Muhl.; this name, therefore, should not be used for this species.

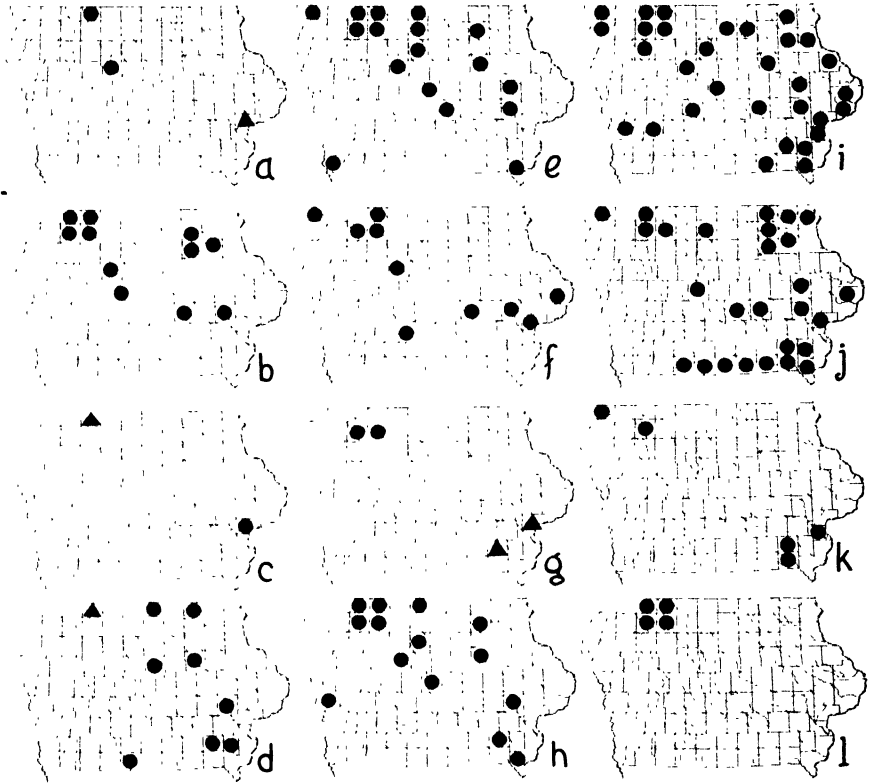


FIGURE 6. Distribution Maps of Iowa Cyperaceae.

- a—*Fimbristylis autumnalis* (▲) and *Eriophorum gracile* (●).
- b—*Eriophorum angustifolium*.
- c—*Bulbostylis capillaris* (●) and *Eleocharis pauciflora* var. *fernaldii* (▲).
- d—*Eleocharis wolffi* (▲) and *Eleocharis tenuis* (●).
- e—*Eleocharis acicularis*.
- f—*Eleocharis compressa*.
- g—*Eleocharis coloradoensis* (●) and *Eleocharis atropurpurea* (▲).
- h—*Eleocharis macrostachya*.
- i—*Eleocharis calva*.
- j—*Eleocharis obtusa*.
- k—*Eleocharis obtusa* var. *engelmanni*.
- l—*Rhynchospora capillacea*.

5. *Eleocharis tenuis* (Willd.) Schultes, Mant. 2: 89. 1824.

*E. capitata* var. *verrucosa* Sv. Rhodora 34: 202. 1932.

*E. tenuis* var. *verrucosa* (Sv.) Sv. Rhodora 41: 66. 1939.

Slender spike-rush—usually in ditches or along muddy stream banks, sometimes in swamps or on wet prairies—Nova Scotia to Maine, Pennsylvania, Indiana, Iowa, and eastern Oklahoma, south to South Carolina, Tennessee, Missouri, Louisiana, and Texas.

IOWA DISTRIBUTION: map 6-d. UNCONFIRMED COUNTY REPORTS: Mahaska (96), Muscatine (99), and Scott (99). NOTES: (a) A report of this species from Emmet County (168) is based on a misidentification of a mixture of *E. compressa* and *E. calva*. The reports from Lyon County (107, 153) are based on misidentifications of *E. calva*, and a report for Poweshiek County (107) was based on a misidentification of *E. compressa*. (b) Svenson (72, 76) considers the Mississippi Valley plants (his var. *verrucosa*) as distinct from the plants of the Eastern Coastal Plain. In my opinion, this variety cannot be separated satisfactorily from the typical form of the species.

6. *Eleocharis coloradoensis* (Britt.) Gilly, Amer. Midl. Nat. 26: 66. 1941.

"*E. pygmaea*," in part, of some authors; not of Torr. 1836 (R).

"*Scirpus nanus*," in part, of some authors; not of Spreng. 1813 (B, BB2, G).

*Scirpus coloradoensis* Britt. Torreyia 4: 93. 1904.

*E. leptos* (Steud.) Sv. Rhodora 31: 176, in part. 1929; not *E. leptos* C. B. Clarke 1900.

*E. parvula* var. *anachaeta* (Torr.) Sv. Rhodora 36: 386, in part. 1934.

Dwarf spike-rush—sandy shores or boggy soil, frequently in alkaline areas—northwestern Iowa and South Dakota to Idaho and southern Nevada, south to Colorado, New Mexico, and southern California.

IOWA DISTRIBUTION: map 6-g. SPECIMENS EXAMINED: Clay County, Round Lake, August 30, 1936, *Hayden* 690 (ISC, NY). Palo Alto County, Freedom Township, Medium Lake, August 18, 1940, *Hayden* 8291 (ISC). UNCONFIRMED COUNTY REPORT: Woodbury (71).

7. *Eleocharis atropurpurea* (Retz.) Kunth, Enum. Pl. 2: 151. 1837.

Purple spike-rush—usually in wet sandy soil—along coastal plain from Georgia to Florida, Texas, and the east coast of Mexico, northward in the Mississippi Valley to Illinois, Iowa, and Nebraska; reported from Colorado; in Italy, Switzerland, and the tropics of both hemispheres.

IOWA DISTRIBUTION: map 6-g. SPECIMENS EXAMINED: Jefferson County, Center Township, September 3, 1933, *McDonald* 1206 (P). Muscatine County, 1895, *Reppert* (NY).

8. *Eleocharis macrostachya* Britt. in Small, Fl. S. E. U. S. 184, 1327. 1903.

"*E. palustris*" of most Iowa authors; not of R. & S. 1817.

"*E. palustris* var. *vigens*" of some authors; not of L. H. Bailey 1889.

"*E. mamillata*" of some American authors; not of Lindb. f. 1902.

"*E. smallii*" of some authors; not of Britt. 1903.

Pale spike-rush—wet soil, swamps, marshes, and along ponds and lakes—Illinois and Iowa to North Dakota, Alberta, and British Columbia, south to Missouri, Oklahoma, Texas, central Mexico, and California; reported from South America.

IOWA DISTRIBUTION: map 6-h. UNCONFIRMED COUNTY REPORTS: Boone (115), Cerro Gordo (141, 142), Decatur (107, 119), Dubuque (145), Fayette (126), Hamilton (107, 137, 141, 142), Hardin (148), Lyon (152, 153), Mahaska (96), Muscatine (99), Scott (99, 107), Shelby (107, 119), Winneshiek (118, 142, 155), and Worth (141, 142). NOTES: (a) Some of these unconfirmed reports, particularly the earlier ones as "*E. palustris*," may refer to *E. calva*. (b) I am unable to separate middle western plants with more acute scales and firmer culms, identified as *E. smallii* by several workers, from the typical form of this species because of a complex series of intergradations. Additional study in the field is needed to determine whether such firm-culmed material represents a variety or is merely an ecotype of *E. macrostachya*. (c) *E. smallii*, the type specimen of which is badly infested with smut, apparently is confined to the Atlantic Coast Region and perhaps is not really separable from other species of the "*palustris*-complex." Fernald and Brackets (35) and Svenson (76) have discussed this complex in considerable detail.

9. *Eleocharis calva* Torr. Fl. N. Y. 2:346. 1843.

"*E. palustris* var. *glaucescens*" of many American authors; not of A. Gray 1867.

"*E. glaucescens*" of some American authors; not of Schultes 1824.

*E. palustris* var. *calva* (Torr.) A. Gray, Man. 522. 1848.

Spathiform spike-rush—in ditches swamps, low wet spots on the prairies, and along muddy stream banks, pond, and lake shores—Nova Scotia to James Bay, Manitoba and Colorado, south to Virginia, Tennessee, northern Arkansas, and New Mexico.

IOWA DISTRIBUTION: map 6-i. NOTES: (a) See the first note under *E. macrostachya*. (b) According to Fernald and Brackets (35) and Svenson (76), *Scirpus glaucescens* Willd., on which both *E. palustris* var. *glaucescens* and *E. glaucescens* are based, is conspecific with the true *E. palustris* of Europe and northeastern North America.

10. *Eleocharis obtusa* (Willd.) Schultes, Mant. 2:89. 1824.

*Scirpus obtusus* Willd. Enum. Pl. Hort. Berol. 1:76. 1809.

"*E. ovata*" of many American authors; not of R. & S. 1817 (BB).

*E. ovata* var. *obtusa* (Willd.) Kük. in Skottsbl. Göteborg. Bot. Trädg. 2:212. 1926.

Blunt spike-rush—commonly found in wet muddy places, sometimes in sandy soil—Nova Scotia and Quebec to Ontario, Minnesota, Nebraska, and Colorado, south to Georgia, western Florida, Louisiana, Texas, and New Mexico; also on the west coast from British Columbia to northern California and inland to eastern Washington; also in the Hawaiian Islands.



**IOWA DISTRIBUTION:** map 6-j. **UNCONFIRMED COUNTY REPORTS:** Dickinson (127, 145, 157), Emmet (145), Madison (170), Mahaska (96), Plymouth (93), Scott (99), and Story (170). **NOTES:** The true *E. ovata* R. & S., in which the tubercle is considerably narrower than the body of the achene, is found in Europe and locally through northern North America (south to New England, Michigan, Minnesota, and in British Columbia) and is closely related to this species. Future study may even disclose that they are not adequately distinct. Extreme forms of *E. obtusa*, with a flattened and lower tubercle and usually longer spikelets, are apparently distinct if one only casually examines them. They are generally recognized as *E. engelmanni*. Many specimens throughout the range of the species, however, are intermediate between the two extremes in relative height of tubercle and achene, in length of perianth bristles, and in shape of achene. I have found achenes referable to *E. obtusa* and others referable to *E. engelmanni* in the same spikelet on several specimens. In view of this intergradation throughout the range of the species, I am unwilling to give them both specific rank; I am, therefore, including the *engelmanni* phase in this paper as a variety. Another variant of the species, almost wholly confined to southern Arkansas and adjacent Texas, is the form with longer spikelets and narrower, more acute scales usually called *E. lanceolata*.<sup>18</sup> Forms without perianth bristles are not infrequent through the range of the species, and unusually robust forms with larger achenes and tubercles are not uncommon. Intermediate specimens have been referred to the species rather than to the following variety.

10a. *Eleocharis obtusa* (Willd.) Schultes, var. *engelmanni* (Steud.) Gilly, comb. nov.

*E. engelmanni* Steud. Syn. Pl. Cyp. 79. 1855.

*E. ovata* var. *engelmanni* (Steud.) Britt. Jour. N. Y. Micr. Soc. 5: 103. 1889.

Engelmann's spike-rush—habitat essentially the same as that of the species—Maine, southern New York, southern Michigan to Iowa, South Dakota, North Dakota, Saskatchewan, and Washington, south to Virginia, Tennessee, Alabama, Arkansas, central Texas, New Mexico, Arizona, and California.

**IOWA DISTRIBUTION:** map 6-k.

#### SPECIES OF ELEOCHARIS DOUBTFULLY OR ERRONEOUSLY REPORTED FOR IOWA

*Eleocharis elliptica* Kunth—The report of this species for Emmet County (128) is based on a specimen which is a mixture of *E. calva* Torr. and *E. compressa* Sull.

*Eleocharis intermedia* (Muhl.) Schultes—This species has been reported from the following counties: Dickinson (124, 128), Floyd (74,

<sup>18</sup> The following transfer seems necessary: *Eleocharis obtusa* (Willd.) Schultes, var. *lanceolata* (Fern.) Gilly, comb. et stat. nov., based on *Eleocharis lanceolata* Fern. Proc. Amer. Acad. 34:493. 1899.

107), Hardin (148), Story (74, 107, 128), and Winneshiek (124). The Dickinson County and Floyd County reports are based on misidentifications of specimens of *E. calva* Torr., and the Story County reports are based on specimens of *E. macrostachya* Britt. I have not seen specimens to substantiate the other reports, but suspect that they, too, are based upon misidentifications. This species also is listed for Iowa in "Gray's" *Manual* (66), Rydberg's *Flora* (67), Britton's *Manual* (13, 14), and the *Illustrated Flora* (15, 16), but I have been unable to find specimens to substantiate such listing. The known range of this species includes Minnesota, Wisconsin, and Illinois, and it is possible that it eventually will be found in northeastern Iowa.

*Eleocharis olivacea* Torr.—A report of this species from Jefferson County (123) is based on a misidentification of a specimen of *E. atropurpurea* (Retz.) Kunth.

#### 9. RHYNCHOSPORA Vahl, Enum. 2:229. 1896."

This genus, in which about 200 species are accepted as distinct, has its center of distribution in the sub-tropics from whence it has spread into both the tropics and the temperate zones. Only one species is definitely known from Iowa.

##### 1. *Rhynchospora capillacea* Torr. Comp. 41. 1826.

Beaked-rush—a species of hanging bogs, prairie sloughs, and wet swamps—Maine to Ontario and Minnesota, south to New Jersey, Pennsylvania, Michigan, and northern Iowa.

IOWA DISTRIBUTION: map 6-1. NOTE: Gale (38) has recently discussed this and related species.

#### 10. SCLERIA Bergius, Königl. Acad. Sv. Handl. 26:142. 1765.

About 200 species are recognized, most of them being in the tropics. Only the following two species have been found in Iowa; these may be readily distinguished from all other Iowa Cyperaceae by their conspicuous, hard, white, shining achenes, each surrounded by several scales.

#### KEY TO THE IOWA SPECIES

- A. Coarse, triangular-stemmed plants, 4 dm.-9 dm. tall; leaves 3 mm.-5 mm. wide; inflorescence usually a terminal cluster; achene 2 mm. long, smooth-surfaced; basal disk (hypogynium) granular-roughened.....1. *S. triglomerata*
- AA. Slender, filiform-stemmed plants, 1 dm.-5 dm. tall; leaves less than 1 mm. wide; inflorescence of 4-6 separate few-flowered clusters; achene 1 mm. long, roughened with reticulated ridges; basal disk lacking .....2. *S. verticillata*

##### 1. *Scleria triglomerata* Michx. Fl. Bor.-Amer. 2:168. 1803.

Tall nut-rush—in wet prairies, along stream banks, frequently in sandy soil—Massachusetts to Ontario, Wisconsin, and Iowa, south to Florida and Texas.

"Although this generic name is sometimes written "*Rhynchospora*," the spelling as used in this paper has been placed on the conserved list of genera and should, therefore, be used.

IOWA DISTRIBUTION: map 7-a. UNCONFIRMED COUNTY REPORTS: Cedar (163), Floyd (107), Johnson (19, 107, 163, 164), Monroe (107), and Muscatine (99, 107, 163).

2. *Scleria verticillata* Muhl. ex Willd. Sp. Pl. 4:317. 1805.

Low nut-rush—bogs, hanging bogs, sloughs, wet meadows, occasionally in sandy soil on the prairies—Connecticut to Ontario and Minnesota, south to Florida, Louisiana, and Texas; also in the West Indies and northern South America.

IOWA DISTRIBUTION: map 7-a. SPECIMENS EXAMINED: Emmet County, Emmet Township, August 27, 1929, *Wolden 1407* (ISC, W), July 14, 1931, *Wolden 1497* (G, ISC), August 4, 1934, *Hayden 220* (ISC); Estherville, September 11, 1934, *Fults 2919* (ISC).

11. CAREX L. Sp. Pl. 972. 1753.

This is the largest genus in the family and the center of its distribution is in the north temperate zone. Between 800 and 1,000 species are recognized. A few more than 500 of these are reported for North America (53). Only 87 species, 7 of which are represented by more than one variety, have been found in Iowa.

For convenience, and to simplify the identification of Iowa *Carex* specimens, it has seemed advisable to break the genus into artificial GROUPS. Separate keys and discussion for the species in each group follow the Key to the Groups.

KEY TO THE GROUPS OF IOWA CAREX

- A. Spikes all, or some of them, gynecandrous (with pistillate flowers at apex and staminate flowers at base).
- B. Beaks of perigynia prominently bidentate at apices (Fig. 3-Q).
- C. Stigmas 2; achenes lenticular or flattened; perigynia flattened, closely surrounding each achene, conspicuously 2-edged and wing-margined (Fig. 3-R; the wing-margin scarcely noticeable in species No. 1, *C. deweyana*) ..... GROUP A (page 96)
- CC. Stigmas 3; achenes triangular or sub-terete in cross-section; perigynia more or less triangular-inflated or somewhat flattened (Fig. 3-T), never, however, wing-margined ..... GROUP B (page 101)
- BB. Beaks of perigynia entire or somewhat obliquely cut at apices (in the latter case, the beaks sometimes minutely bidentulate at maturity—Fig. 3-S) ..... GROUP C (page 102)
- AA. Spikes all unisexual (either staminate or pistillate) or some of them androgynous (with staminate flowers at apex and pistillate flowers at base).
- B. Beaks of the perigynia entire or somewhat obliquely cut at apices (in the latter case, the beaks sometimes minutely bidentulate at maturity—Fig. 3-S).
- C. Stigmas 3; achenes triangular or subterete; perigynia triangular or rounded in cross-section (Fig. 3-T) ..... GROUP D (page 104)
- CC. Stigmas 2; achenes lenticular or flattened; perigynia flattened, 2-edged or more or less wing-margined (at least above the middle—Fig. 3-Q, R) ..... GROUP E (page 111)
- BB. Beaks of the perigynia prominently bidentate at apices (Fig. 3-Q).
- C. Stigmas 2; achenes lenticular or flattened; perigynia flattened and 2-edged (at least above the middle), the lower portion of the body sometimes spongy-inflated or enlarged ..... GROUP F (page 116)
- CC. Stigmas 3; achenes triangular or subterete in cross-section; perigynia obtusely triangular or terete in cross-section (somewhat flattened in species No. 69, *C. sprengelii*), either closely surrounding the achene or loose and inflated, but not spongy-based ..... GROUP G (page 122)

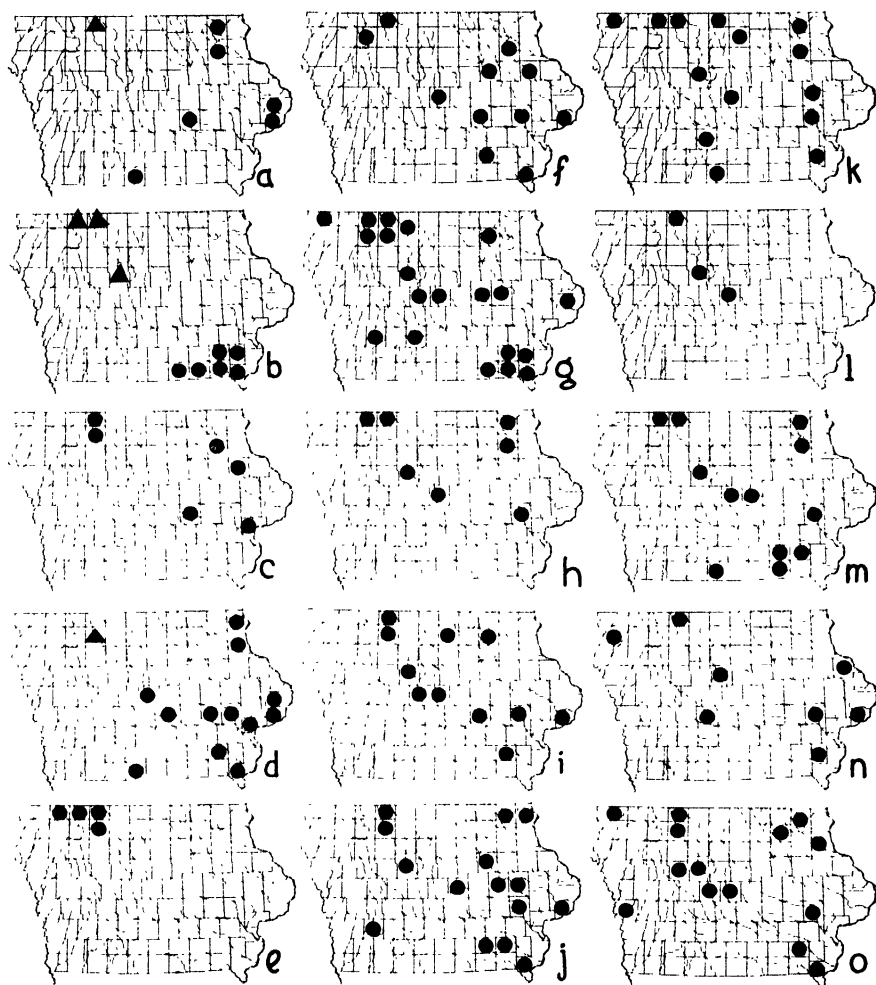


FIG. 7. Distribution Maps of Iowa Cyperaceae.

FIGURE 7. Distribution Maps of Iowa Cyperaceae.

- a—*Scleria verticillata* (▲) and *Scleria triglomerata* (●).
- b—*Carex deweyana* (▲) and *Carex squarrosa* (●).
- c—*Carex interior*.
- d—*Carex foenea* (▲) and *Carex muskingumensis* (●).
- e—*Carex sychnocephala*.
- f—*Carex tribuloides*.
- g—*Carex cristatella*.
- h—*Carex projecta*.
- i—*Carex suberecta*.
- j—*Carex scoparia*.
- k—*Carex bebbii*.
- l—*Carex tenera*.
- m—*Carex normalis*.
- n—*Carex festucacea*.
- o—*Carex molesta*.

KEY TO IOWA CAREX—GROUP A<sup>20</sup>

- A. Perigynia conspicuously spongy-thickened at the base, the wing-margin scarcely (if at all) noticeable; pistillate scales tipped with a scabrid awn half the length of the scales ..... 1. *C. deweyana*
- AA. Perigynia not spongy-thickened at the base, the wing-margin evident (although narrow in a few species); pistillate scales obtuse or acute at apices, never scabrid-awned.
  - B. Spikes loosely few-flowered, the perigynia radiating in all directions; pistillate scales suborbicular or obovate, broadest at or above the middle ..... 2. *C. interior*
  - BB. Spikes compactly few- to numerous-flowered, the perigynia ascending or somewhat spreading, but not widely radiating; pistillate scales lanceolate, oblong, or ovate, generally broadest below the middle.
    - C. Culms arising solitary or few together from long creeping rhizomes; some of the spikes sometimes entirely staminate or nearly so... 3. *C. foenea*
  - CC. Culms loosely to densely caespitose, rhizomes (if present) very short; spikes all gynecandrous or pistillate, none completely staminate.
    - D. Inflorescence bracts (at least several of the lowermost) leaf-like; conspicuous, considerably longer than the inflorescence; inflorescence a capitate head of numerous close-crowded spikes, the individual spikes scarcely distinguishable ..... 4. *C. sychnocephala*
    - DD. Inflorescence bracts scale-like, or some of the lowermost setaceous, never leaf-like nor conspicuously exceeding the inflorescence; inflorescence a more or less capitate cluster of spikes (but the individual spikes distinguishable) or the spikes more or less separated and distant.
  - E. Ventral side of leaf-sheaths (Fig. 3-O) green, strongly nerved and similar in texture to the dorsal side, with more or less of a V- or Y-shaped hyaline area at the mouth, or with a narrow hyaline area down the center of the otherwise green ventral side.
  - F. Perigynia nerved on the ventral surface (Fig. 3-P).
    - G. Perigynia lanceolate, one-third or less than one-third as wide as long.
    - H. Marginal wings of the perigynia broad above, somewhat constricted or narrowed at about the middle of the perigynium body, the narrowed margin continuing to the base.
      - I. Spikes 1.5 cm.–2.5 cm. long; perigynia 7 mm.–10 mm. long ..... 5. *C. muskingumensis*
    - II. Spikes less than 1.5 cm. long; perigynia less than 7 mm. long.
      - J. Leaf-sheaths completely green dorsally (Fig. 3-O); culms stiff; leaf blades firm; spikes aggregated into a compact head.
        - K. Perigynia thin and scale-like except where distended over achene, their tips appressed or ascending; spikes ovoid-oblong or sub-globose, 6 mm.–12 mm. long, 4 mm.–8 mm. in diameter ..... 6. *C. tribuloides*
      - KK. Perigynia thickened plano-convex, their tips widely spreading or recurved; spikes almost globose, 4 mm.–8 mm. in length and diameter ..... 7. *C. cristatella*
    - JJ. Leaf-sheaths green and white mottled or white hyaline between the green nerves on the dorsal side (Fig. 3-O); culms more or less flexible and lax; leaves flaccid; spikes more or less separated in a lax moniliform inflorescence. 8. *C. projecta*

<sup>20</sup> Species 4 to 17, inclusive, are placed by Mackenzie (53, 54) in the Section *Ovales*, a section comprising one of the most difficult species-groups of the genus *Carex*. I believe that Mackenzie, and most other modern workers as well, have recognized entirely too many species in this section of the genus. Because of variation among individuals of single colonies, the value of certain characters—(a) more or less green *vs.* hyaline ventral surface of leaf sheath; (b) green and white mottled *vs.* entirely green dorsal surface of leaf sheath; and (c) nerveless *vs.* more or less nerved dorsal and ventral surfaces of the perigynia—for the identification of and the recognition of species well may be questioned. The variability in degree of aggregation of spikes in the inflorescence presents equal problems in delimiting species. Herbarium study alone will not solve these problems. Several years of intensive field study and collecting, combined with herbarium and transplant studies, will be necessary to accurately determine (if this is at all possible) the extent of variability within a species in this section of the genus. It is almost certain that eventual re-evaluation of the Section *Ovales* will result in an entirely different treatment of the group.

- HH. Marginal wings of the perigynia narrow for their entire length ..... 10. *C. scoparia*
- GG. Perigynia suborbicular or orbicular, at least one-half as wide as long.
  - H. Perigynia 3 mm.-4 mm. long, 1.5 mm.-2 mm. broad; spikes loosely aggregated or the inflorescence moniliform 13. *C. normalis*
  - HH. Perigynia 5.5 mm.-6.5 mm. long, 2.75 mm.-4 mm. broad; spikes closely aggregated ..... 17. *C. bicknellii*
- FF. Perigynia nerveless on the ventral surface (Fig. 3-P).
  - G. Wing-margin broad to the base; perigynia 4 mm.-5 mm. long, 2.25 mm.-2.75 mm. broad ..... 9. *C. suberecta*
  - GG. Wing-margin narrow for entire length; perigynia 3 mm.-3.5 mm. long, 1.5 mm.-2 mm. broad ..... 11. *C. bebbii*
- EE. Ventral surface of leaf-sheaths (Fig. 3-O), or at least the greater portion of their width, hyaline.
  - F. Perigynia subulate to narrowly ovate-lanceolate, three to four times as long as broad; marginal wings of perigynia narrow for their entire length ..... 10. *C. scoparia*
  - FF. Perigynia ovate-lanceolate, ovate or orbicular, never more than twice as long as broad; marginal wings of the perigynia broad for their entire length (except in species No. 8, *C. projecta*, where the wing is narrowed about the middle of the perigynium body).
    - G. Perigynia ovate-lanceolate or narrowly ovate, 3 mm.-4 mm. (or, rarely, 5 mm.) long
      - H. Dorsal surface of leaf-sheaths completely green.
        - I. Spikes aggregated into a compact head; perigynia brown or brownish at maturity, the ventral surface nerveless ..... 11. *C. bebbii*
        - II. Spikes loosely arranged in a moniliform inflorescence; perigynia straw-colored at maturity, the ventral surface nerved (at least at base) ..... 12. *C. tenera*
      - HH. Dorsal surface of leaf-sheaths green and white mottled or white hyaline between the green nerves.
        - I. Perigynia 3 mm.-4 mm. long, 1.5 mm.-2 mm. broad; inflorescence moniliform or the spikes loosely aggregated ..... 13. *C. normalis*
        - II. Perigynia 3.25 mm.-5 mm. long, 1.5 mm. (or less) broad; inflorescence always moniliform ..... 8. *C. projecta*
    - GG. Perigynia suborbicular or orbicular, 3.5 mm.-6.5 mm. long.
      - H. Spikes more or less separated in a lax, moniliform inflorescence; perigynia 3.5 mm. long, beaks about as long as the bodies; achenes 1.5 mm. long, oblong-ovoid or oblong ..... 14. *C. festucacea*
      - HH. Spikes closely aggregated; perigynia 3.75 mm.-6.5 mm. long; beaks half (or less than half) as long as the bodies; achenes 1.75 mm.-2 mm. long, suborbicular or orbicular.
        - I. Perigynia 3.75 mm.-5 mm. long, firm-textured, thickened plano-convex in cross-section.
          - J. Ventral surface of perigynia nerved; perigynia tapering into beaks; dorsal surface of leaf-sheaths green and white mottled or white-hyaline between the green nerves ..... 15. *C. molesta*
          - JJ. Ventral surface of perigynia nerveless; perigynia abruptly contracted into beaks; dorsal surface of leaf-sheaths entirely green ..... 16. *C. brevior*
        - II. Perigynia 5.5 mm.-6.5 mm. long, membranous, thin except where distended over achene ..... 17. *C. bicknellii*

1. *Carex deweyana* Schw. Ann. Lyc. N. Y. 1: 65. 1824.

Dewey's sedge—woodlands—Labrador and Newfoundland to Mackenzie, south to Pennsylvania, Michigan, Iowa, Colorado, and British Columbia.

IOWA DISTRIBUTION: map 7-b. SPECIMENS EXAMINED: Dickinson County, Spirit Lake, June 21, 1881, *Arthur 967b* (ISC). Emmet County, Ft. Defiance State Park, June 24, 1931, *Wolden 1491* (W). Webster County, Fort Dodge, July 8, 1905, *Oleson* (ISC), June 14, 1906, *Cratty* (ISC).

2. *Carex interior* L. H. Bailey, Bul. Torrey Club 20:426. 1893.  
 "C. *stellulata*" of some Iowa authors; not of Gooden. 1794.  
*C. scirpoides* Schkuhr ex Muhl. Descr. Gram. 225. 1817; not *C. scirpoidea* Michx. 1803 (G).  
*C. stellulata* var. *scirpoides* Carey in A. Gray, Man. 544. 1848.

Inland sedge—wet meadows, boggy spots, hanging bogs, and low wet prairies—Labrador to Newfoundland and British Columbia south to New Jersey, Pennsylvania, Indiana, Kansas, and California; also in northern Mexico.

IOWA DISTRIBUTION: map 7-c. UNCONFIRMED COUNTY REPORT: Scott (99, 107).

3. *Carex foenea* Willd. Enum. 957. 1809.  
*C. siccata* Dewey, Amer. Jour. Sci. 10:278. 1826.

Dry-spiked sedge—dry prairie, in rocky places, and along roadside banks—Labrador and Newfoundland, west to Yukon, south to Connecticut, New York, Michigan, northern Iowa, Montana, and British Columbia. IOWA DISTRIBUTION: map 7-d. SPECIMEN EXAMINED: Clay County, Peterson Township, June, 1936, *Hayden* 652 (ISC, NY). NOTES: (a) A report of this species from Emmet County (94) is based upon a specimen of *C. eleocharis*. (b) Svenson (75) has recently presented evidence, based on examination of the Willdenow herbarium at Berlin, which indicates that the name *C. foenea* should replace *C. siccata*; the "*C. foenea*" of most American authors should be called *C. argyrantha* Tuckerm. (See also page 132 of this paper.)

4. *Carex sychnocephala* Carey, Amer. Jour. Sci. II 4:24. 1847.

Long-beaked sedge—marshes, sloughs, meadows and thickets, lake margins, frequently in sandy soil—Ontario to Saskatchewan, south to New York, Michigan, Iowa, and Montana.

IOWA DISTRIBUTION: map 7-e.

5. *Carex muskingumensis* Schw. Ann. Lyc. N. Y. 1:66. 1824.  
*C. arida* Schw. & Torr. Ann. Lyc. N. Y. 1:312. 1825.

Muskingum sedge—low moist woodlands and thickets on river floodplains—Michigan to Manitoba, south to Kentucky, Missouri, and southern Kansas.

IOWA DISTRIBUTION: map 7-d. UNCONFIRMED COUNTY REPORTS: Dubuque (90) and Poweshiek (107).

6. *Carex tribuloides* Wahl. Sv. Vet.-Akad. Nya. Handl. 24:145. 1803.  
*C. lagopodioides* Schkuhr ex Willd. Sp. Pl. 4:230. 1805.

Blunt broom sedge—wet meadows and low swampy woodlands—Maine to Quebec and Minnesota, south to Florida, Louisiana, and Oklahoma.

IOWA DISTRIBUTION: map 7-f. UNCONFIRMED COUNTY REPORTS: Dickinson (157), Henry (103), Mahaska (97), Muscatine (99), and Winneshiek (124,

155). NOTE: A report of this species from Lee County (107) was based on a misidentification of a specimen of *C. scoparia*.

7. *Carex cristatella* Britt. in Britt. & Brown, Ill. Fl. 1:357. 1896.

*C. cristata* Schw. Ann. Lyc. N. Y. 1:66. 1824; not of Clairv. 1811 (G).

*C. straminea* var. *cristata* (Schw.) Tuckerm. Enum. Caric. 18. 1843.

*C. tribuloides* var. *cristata* (Schw.) L. H. Bailey, Proc. Amer. Acad. 22:148. 1886.

Crested sedge—low ground, marshes, swamps, swales, wet prairies, and woodland-edge thickets—Michigan to Manitoba, south to Kentucky, Missouri, and eastern Kansas.

IOWA DISTRIBUTION: map 7-g. UNCONFIRMED COUNTY REPORTS: Adams (120), Decatur (87, 107, 119), Hardin (148), Johnson (149), Muscatine (99, 107, 159), Scott (99, 107), and Winneshiek (155). NOTE: A report of this species from Calhoun County (150) was based on a misidentification of a specimen of *C. molesta*.

8. *Carex projecta* Mackenz. Bul. Torrey Club 35: 264. 1908.

*C. tribuloides* var. *reducta* L. H. Bailey, Proc. Amer. Acad. 22:148. 1886 (G).

*C. tribuloides* var. *moniliformis* Britt. in Britt. and Brown, Ill. Fl. 1:356. 1896 (B, BB).

Necklace sedge—wet ground and open moist woodlands—Newfoundland to Manitoba, south to Virginia, Michigan, and Iowa; reported from British Columbia.

IOWA DISTRIBUTION: map 7-h. NOTE: A report of this species from Johnson County (107) was based on a misidentification of specimens of *C. bebbii* and *C. normalis*; a report of this species from Story County (107) was based on a misidentification of a specimen of *C. normalis*.

9. *Carex suberecta* (Olney) Britt. Man. ed. 2 1057. 1903.

*C. straminea* var. *ferruginea* L. H. Bailey, Bul. Torrey Club 20:421. 1893.

Prairie straw sedge—low ground in prairies and moist meadows, swales, and along pond edges—Ontario to Minnesota, south to western Virginia, Indiana, and Iowa.

IOWA DISTRIBUTION: map 7-i.

10. *Carex scoparia* Schkuhr ex Willd. Sp. Pl. 4:230. 1805.

Pointed broom sedge—low wet prairie, in open marshes, and frequently along roadsides—Newfoundland to British Columbia, south to South Carolina, Arkansas, Nebraska, and Oregon; also reported from Mexico.

IOWA DISTRIBUTION: map 7-j. UNCONFIRMED COUNTY REPORTS: Dickinson (157), Fayette (117), Floyd (107), Hardin (148), Henry (103), Mahaska (96), and Muscatine (99, 159). NOTE: A report of this species from Lee



County (121) was based on misidentification of a specimen of *C. tribuloides*.

11. *Carex bebbii* Olney ex. Fern. Proc. Amer. Acad. 37:478. 1902.

*C. tribuloides* var. *bebbii* L. H. Bailey, Mem. Torrey Club. 1: 55. 1889 (B, BB).

Bebb's sedge—swampy meadows, prairie sloughs, and marshes—Newfoundland to British Columbia, south to New Jersey, Indiana, Iowa, Colorado, and Washington.

IOWA DISTRIBUTION: map 7-k.

12. *Carex tenera* Dewey, Amer. Jour. Sci. 8:97. 1824.

*C. straminea* var. *tenera* (Dewey) Boott, Ill. Carex 120. 1862.

*C. straminea* var. *echinodes* Fern. Proc. Amer. Acad. 37:474. 1902.

*C. tenera* var. *echinodes* (Fern.) Wiegand, Rhodora 26:2. 1924.

Marsh straw sedge—moist meadows, dry prairies, on banks and sometimes in open woodlands and thickets—New Brunswick to Quebec, and Alberta, south to North Carolina, Indiana, Missouri, and Montana.

IOWA DISTRIBUTION: map 7-l. SPECIMENS EXAMINED: Emmet County, July, 1923, *Wolden* (ISC); Armstrong, June, 1890, *Cratty* (ISC); Estherville Township, August 13, 1934, *Hayden* 196 (ISC, NY). Story County, Ames, 1895, *Carver* (ISC), June 20, 1898, *Ball* (ISC). Webster County, Fort Dodge, June 20, 1910, *Oleson* (ISC). UNCONFIRMED COUNTY REPORTS: Dickinson (107, 108), Fayette (126), Floyd (92), Johnson (107, 164), Lee (92), Mahaska (96), and Winneshiek (155). NOTE: A report of this species from Jasper County (107) was based on a misidentification of a specimen of *C. brevior*; a report from Winnebago County (107) was based on a misidentification of a specimen of *C. bebbii*.

13. *Carex normalis* Mackenz. Bul. Torrey Club 37:244. 1910.

*C. mirabilis* Dewey, Amer. Jour. Sci. 30:63. 1836; not of Host. 1809 (G).

*C. straminea* var. *mirabilis* (Dewey) Tuckerm. Enum. Caric. 18. 1843 (B, BB).

*C. cristata* var. *mirabilis* (Dewey) Boott ex A. Gray, Man. ed. 5 580. 1867.

*C. mirabilis* var. *perlonga* Fern., Proc. Amer. Acad. 37:473. 1902.

Larger straw sedge—dry open woodlands—Maine to Ontario and Manitoba, south to North Carolina, Kentucky, Missouri, and Oklahoma. IOWA DISTRIBUTION: map 7-m. UNCONFIRMED COUNTY REPORTS: Chickasaw (126), Mahaska (96), Poweshiek (107), and Warren (124).

14. *Carex festucacea* Schkuhr ex. Willd. Sp. Pl. 4:242. 1805.

*C. straminea* var. *festucacea* (Schkuhr) Tuckerm. Enum. Caric. 18. 1843.

Fescue sedge—dry or moist prairie and in open, often moist, woodlands—Massachusetts to Indiana and Iowa, south to Georgia and Louisiana.

IOWA DISTRIBUTION: map 7-n. UNCONFIRMED COUNTY REPORTS: Allamakee (160), Decatur (107), Dickinson (107, 157), Floyd (107), Hamilton (107), Hardin (148), Lee (121), Linn (107), Louisa (102), Lucas (107), Monroe (107), Muscatine (102, 156, 159), Polk (107), and Poweshiek (94). NOTES: A report of this species from Black Hawk County (104) is based on a misidentification of a specimen of *C. suberecta*; a report from Floyd County (107) is based on a specimen of *C. brevior*; a report from Harrison County (107) is based on a specimen of *C. molesta*; a report from Johnson County (107) is based on misidentifications of specimens of *C. cristatella* and *C. molesta*; and reports from Story County (94, 107) are based on misidentifications of *C. bicknellii*.

15. *Carex molesta* Mackenz. N. Amer. Fl. 18:151. 1931.

Repulsive sedge—dry and moist prairies or dry woodland—Michigan to Nebraska, south to Indiana, Missouri, and Kansas.

IOWA DISTRIBUTION: map 7-o. NOTE: Only 1 specimen of this species has been previously reported for the state (128) although many of the specimens which I have examined were reported by other workers under other names.

16. *Carex brevior* (Dewey) Mackenz. Bul. Torrey Club 42:605. 1915.

*C. straminea* var. *brevior* Dewey, Amer. Jour. Sci. 11:158. 1862 (BB2).

*C. straminea* var. *typica* Boott, Ill. Carex 121. 1862.

*C. festucacea* var. *brevior* (Dewey) Fern. Proc. Amer. Acad. 37:477. 1902 (G).

Few-headed straw sedge—prairies and open meadows—Maine to Manitoba and British Columbia, south to Virginia, Tennessee, Texas, New Mexico, and Washington.

IOWA DISTRIBUTION: map 8-a. UNCONFIRMED COUNTY REPORT: Mahaska (96). NOTE: A report of this species from Johnson County (162) was based on a misidentification of a specimen of *C. molesta*; a report from Lee County (121) is based on a misidentification of *C. bicknellii*.

17. *Carex bicknellii* Britt. and Britt. & Brown, Ill. Fl. 1:360. 1896.

*C. straminea* var. *crawei* Boott, Ill. Carex 121. 1896.

*C. straminea* var. *meadei* Boott, l. c.

Bicknell's sedge—moist or dry prairies—Massachusetts to Wisconsin and Saskatchewan, south to New Jersey, Indiana, Arkansas, and Oklahoma.

IOWA DISTRIBUTION: map 8-b. UNCONFIRMED COUNTY REPORTS: Decatur (87), Dickinson (157), and Mahaska (96).

KEY TO IOWA CAREX—GROUP B

- A. Spikes thick-cylindrical or subglobose, 1 cm. or more in diameter, erect on stiff peduncles; pistillate flowers (and perigynia) per spike 100 or more, densely crowded together in numerous rows; perigynia abruptly contracted into slender beaks about as long as the perigynium bodies.

- B. Spikes globose or oblong-ovoid, not more than twice as long as their diameter, usually solitary but sometimes 2 or 3 per culm; perigynia beaks widely spreading, the spikes thus appearing strongly bristled; style abruptly bent just above achene ..... 18. *C. squarrosa*
- BB. Spikes oblong-cylindrical, usually more than twice as long as their diameter, rarely solitary; perigynia beaks appressed-ascending, the spikes thus appearing comparatively smooth; style straight for entire length ..... 19. *C. typhina*
- AA. Spikes linear, elongated, at the most only 5 mm.-6 mm. in diameter, widely spreading or drooping on slender peduncles; pistillate flowers (and perigynia) per spike 10-40, loosely arranged in few rows; perigynia gradually tapering into more or less oblique beaks less than one-fourth as long as the perigynium bodies ..... forms of 20. *C. davisii* (in Group C)

18. *Carex squarrosa* L. Sp. Pl. 973. 1753.

Squarrose sedge—swampy or wet woodlands and floodplain swamps—western Connecticut and western Quebec to Wisconsin, Iowa, and Nebraska, south to North Carolina, Tennessee, and Arkansas.

IOWA DISTRIBUTION: map 7-b. UNCONFIRMED COUNTY REPORTS: Clayton (118) and Dubuque (118). NOTE: The report of this species from Jasper County (107) was based on a misidentification of a specimen of *C. typhina*.

19. *Carex typhina* Michx. Fl. Bor.-Amer. 2:169. 1803.

*C. typhinoides* Schw. Ann. Lyc. N. Y. 1:66. 1824 (B, BB, G).

Cat-tail sedge—wet alluvial woodlands, swamps, marshes, and along river banks—western Massachusetts and western Quebec to Wisconsin and Iowa, south to South Carolina, Kentucky, and Louisiana.

IOWA DISTRIBUTION: map 8-c. UNCONFIRMED COUNTY REPORTS: Clinton (107), Henry (103), and Muscatine (99, 107).

KEY TO IOWA CAREX—GROUP C

- A. Pistillate scales shorter than or scarcely exceeding perigynia, obtuse, or merely acute, or the midribs prolonged into short glabrous mucros; stigmas 3.
- B. Perigynia obtusely triangular or nearly round in cross-section, conspicuously several- to many-nerved or ribbed; spikes sessile or peduncled.
- C. Spikes linear, elongated; rather loosely-flowered and lax, at least the lower ones drooping on slender peduncles.
- D. Leaf blades and sheaths conspicuously pubescent; perigynia 4 mm.-5 mm. long, 2 mm.-2.5 mm. in diameter, short-beaked, the beak obliquely cut or bidentulate ..... 20. *C. davisii*
- DD. Leaf blades glabrous, the sheaths essentially so; perigynia 2.5 mm.-3.5 mm. long, 1.5 mm. in diameter, beakless ..... 21. *C. gracillima*
- CC. Spikes oblong-cylindrical, stiff, closely numerous-flowered, sessile or erect on short peduncles.
- D. Sheaths and leaf blades conspicuously pubescent; pistillate scales hyaline with green midribs.
- E. Perigynia about 2 mm. long, more or less flattened on the side toward the axis of the spike; pistillate scales ovate-triangular, acute or acuminate or obtuse, shorter than the perigynia ..... 22. *C. hirsutella*
- EE. Perigynia 2.5 mm.-3.5 mm. long, nearly round in cross-section; pistillate scales triangular-lanceolate, long-acuminate, cuspidate, or awned, longer than the perigynia ..... 23. *C. bushii*
- DD. Sheaths and leaf blades glabrous, the latter somewhat rough-margined; pistillate scales purple-brown or blackish with light-colored midribs ..... 24. *C. burxbaumii*
- BB. Perigynia more or less flattened, narrowly 2-keeled, otherwise nerveless, abruptly contracted into minute but evident beaks; spikes, at least the lower, on long flexuous peduncles ..... 25. *C. shortiana*

- AA. Pistillate scales with midribs prolonged into scabrous awns; stigmas 3 or 2.  
 B. Stigmas 3; perigynia nearly terete in cross-section, numerous-nerved; spikes sessile to long-peduncled, erect; awns of the pistillate scales shorter than or only slightly exceeding the perigynia in length ..... abnormal forms of 40. *C. blanda* (in Group D)  
 BB. Stigmas 2; perigynia flattened, 2-edged, otherwise nerveless; spikes drooping on flexuous peduncles; awns of the pistillate scales greatly exceeding the perigynia in length ..... forms of 55. *C. crinita* (in Group E)

20. *Carex davisii* Schw. & Torr. Ann. Lyc. N. Y. 1:326. 1825.

Davis' sedge—alluvial woodlands, on floodplains, and in wet ravines—Vermont to Minnesota, south to Maryland, Tennessee, Missouri, and Kansas, west to Oklahoma and Texas.

IOWA DISTRIBUTION: map 8-d. UNCONFIRMED COUNTY REPORTS: Hardin (148), Henry (103), Jasper (107), Muscatine (99, 107), and Scott (99, 107).

21. *Carex gracillima* Schw. Ann. Lyc. N. Y. 1:66. 1824.

Graceful sedge—low moist woodlands and thickets—Newfoundland to Manitoba, south to Virginia, Kentucky, and Missouri.

IOWA DISTRIBUTION: map 8-e. SPECIMENS EXAMINED: Chickasaw County, Williamstown, July 11, 1926, *Spiker* (ISC). Emmet County, Estherville, June 9, 1923, *Wolden* 778 (ISC, W).

22. *Carex hirsutella* Mackenz. Bul. Torrey Club 50:349. 1923.

*C. triceps* var. *hirsuta* (Willd.) L. H. Bailey, Mem. Torrey Club 1:35. 1889 (G).

Hirsute sedge—rocky woodlands, dry copses, and meadows—Maine to southern Ontario, Michigan, Iowa, and Oklahoma, south to South Carolina, Alabama, and Texas.

IOWA DISTRIBUTION: map 8-e. NOTE: Previously reported from Lee County (121) as "*Carex hirsutula*."

23. *Carex bushii* Mackenz. Bul. Torrey Club 37:241. 1910.

Bush's sedge—sandy soil along streams, open woodlands, dry meadows, and roadsides—Massachusetts to Michigan, Illinois, Iowa, and Kansas, south to Virginia, Mississippi, and Texas.

IOWA DISTRIBUTION: map 8-f. SPECIMENS EXAMINED: Davis County, Lick Creek Township, near Floris, June 23, 1939, *Hayden* 9055 (ISC). Wapello County, Richland Township, 5 miles north of Wapello, June 22, 1939, *Hayden* 9029 (ISC). NOTE: This seems to be the first report of this species for the state.

24. *Carex buxbaumii* Wahl. Sv. Vet.-Akad. Nya. Handl. 24:163. 1803.

"*C. fusca*" of many authors; not of All. 1785 (B).

*C. polygama* Schkuhr, Riedgr. 84. 1801; not of J. F. Gmel. 1791 (G).

Buxbaum's sedge; brown sedge—low ground, sloughs, marshes, swamps, and wet meadows—Newfoundland to Ontario, Manitoba, British Columbia, and Alaska, south to Georgia, Arkansas, Colorado, and California.

IOWA DISTRIBUTION: map 8-g. UNCONFIRMED COUNTY REPORTS: Hardin (148) and Scott (99, 107).

25. *Carex shortiana* Dewey, Amer. Jour. Sci. 30:60. 1836.

Short's sedge—marshes, low wet prairies, moist woodlands, and thickets—Pennsylvania to southern Ontario, Indiana, and Iowa, south to Virginia, Tennessee, Missouri, and eastern Kansas.

IOWA DISTRIBUTION: map 8-h.

KEY TO IOWA CAREX—GROUP D

- A. Inflorescence composed of several to numerous spikes.
  - B. Staminate scales truncated and erose at apices, their margins united at the base, each scale surrounding the base of the next above; staminate spikes few-flowered ..... 26. *C. jamesii*
- BB. Staminate scales obtuse or acute at apices, their margins free to the base, the scales spirally imbricated and merely overlapping those above; staminate spikes few- to numerous-flowered.
  - C. Bracts of the inflorescence consisting merely of bladeless sheaths or (at the most) with very short and rudimentary blades.
    - D. Pistillate scales obtuse or acute, the midribs not extending to the apices of the scales; inflorescence bracts bladeless.
      - E. Staminate spikes inconspicuous, 4mm.-8 mm. long, sessile or almost so, surpassed in length by the upper pistillate spikes; perigynia glabrous; pistillate scales rounded at apices, about one-half as long as the perigynia ..... 27. *C. eburnea*
    - EE. Staminate spikes conspicuous, 10 mm.-25 mm. long, long-peduncled and exceeding the pistillate spikes; perigynia appressed-pubescent; pistillate scales acute at apices, longer than the perigynia and almost completely covering them ..... 28. *C. richardsonii*
  - DD. Pistillate scales with midribs prolonged into conspicuous mucros; inflorescence bracts with short and rudimentary blades. . . . 29. *C. pedunculata*
- CC. Bracts of the inflorescence with conspicuous and leaf-like blades.
  - D. Beaks of the perigynia obsolete, minute or very short and tubular, entire, never more than one-fourth as long as the bodies of the perigynia; perigynia glabrous or granular-roughened, fusiform or ovoid.
    - E. Pistillate scales broader than, and as long or longer than the perigynia; pistillate spikes drooping on slender, flexuous peduncles ..... 30. *C. limosa*
  - EE. Pistillate scales not broader and longer than the perigynia; pistillate spikes erect or somewhat spreading, but not drooping.
    - F. Perigynia tapering into conspicuous but short tubular beaks; pistillate scales with midribs prolonged into scabrid awns as long as or longer than the perigynia.
      - G. Sheaths of leaves and bracts glabrous; ligule prolonged beyond base of blade, conspicuous ..... 31. *C. oligocarpa*
    - GG. Sheaths of leaves and bracts quite pubescent; ligule short, truncated and inconspicuous ..... 32. *C. hitchcockiana*
  - FF. Perigynia beakless or very minutely beaked (if the beak appears to be tubular the scales are not long-awned); pistillate scales obtuse, acute or mucronate.
    - G. Culms sharply or obtusely triangular, neither flattened nor conspicuously wing-margined (some specimens of species No. 34, *C. meadii*, are obscurely winged on one angle of the culm); beaks of perigynia erect or somewhat oblique (but not conspicuously bent or recurved) or obsolete.
    - H. Perigynia with numerous nerves, the intervals between the nerves several times wider than the thickness of the nerves.
      - I. Nerves of the perigynia prominently raised above the rest of the perigynium surface.

- J. Staminate spikes conspicuous, long-peduncled, much exceeding the uppermost pistillate spike; pistillate scales about two-thirds as long or as long as the perigynia.
- K. Perigynia glabrous, broadest at the middle, tapering equally to both base and apex, yellowish or green when mature.
  - L. Pistillate spikes rather loosely flowered, 3.5 mm.-5 mm. thick; culms slender; leaves bright green; perigynia dark green when mature ..... 33. *C. tetanica*
  - LL. Pistillate spikes closely flowered, 5 mm.-7 mm. thick; culms stoutish; leaves gray-green; perigynia yellowish-green when mature ..... 34. *C. meadii*
- KK. Perigynia minutely granular-roughened or papillose, broadest near the rounded base, tapering toward the apex, reddish-brown when mature ..... 35. *C. crawei*
- JJ. Staminate spike inconspicuous, sessile or very short-peduncled, not at all or only slightly exceeding the uppermost pistillate spike; pistillate scales one-half as long, or less than one-half as long, as the perigynia ..... 36. *C. granularis*
- II. Nerves of the perigynia impressed, lower than the remainder of the perigynium surface ..... 37. *C. conoidea*
- HH. Perigynia with many fine nerves, the intervals between the nerves scarcely more than twice the thickness of the nerves.
  - I. Staminate spike sessile or very short-peduncled, scarcely (if at all) exceeding the uppermost pistillate spike; perigynia 4.5 mm.-5.5 mm. long, 2 mm.-2.5 mm. wide, glabrous, somewhat turgid-inflated; pistillate spikes closely 4-15 flowered, the upper two pistillate spikes approximate ..... 38. *C. amphibola*  
var. *turgida*
  - II. Staminate spikes on short or long peduncles, exceeding the uppermost pistillate spike; perigynia 2.75 mm.-3.5 mm. long, 1.5 mm. wide, minutely hispidulous or glabrate, closely surrounding the achene and not at all inflated; pistillate spikes loosely 3-8 flowered, usually well separated, the uppermost two not approximate ..... 39. *C. laxiculmis*
- GG. Culms obtusely triangular or much-flattened, conspicuously wing-margined on two of the angles; beaks of the perigynia abruptly bent or recurved.
  - H. Leaves of the flowering culms 3 mm.-10 mm. broad, those of the sterile culms scarcely (if at all) broader; pistillate scales oblong, the midribs prolonged into conspicuous scabrid awns ..... 40. *C. blanda*
  - HH. Leaves of the flowering culms 7 mm.-15 mm. broad, those of the sterile culms conspicuously lanceolate, 12 mm.-30 mm. broad; pistillate scales obovate, acute or bluntish at apices ..... 41. *C. albursina*
- DD. Beaks of perigynia abruptly contracted or tapering, strongly obliquely cut at apices, from one-third as long to longer than the bodies of the perigynia; perigynia short-pubescent, at least at the base of the beak.
  - E. Beaks of perigynia tapering, as long as or longer than the body of the perigynia; leaf blades and sheaths glabrous ..... 42. *C.assiniboinensis*
  - EE. Beaks of perigynia rather abruptly contracted, one-third to one-half as long as body of perigynium; leaf blades and sheaths pubescent ..... 43. *C. hirtifolia*
- AA. Inflorescence of a single terminal androgynous spike; perigynia ellipsoid, with numerous nerves, beakless, yellowish or brown and shining when mature; leaves 0.5 mm.-1.25 mm. wide; culms to at least 6 dm. tall. . . . (*C. leptalea*; see page 133)

26. *Carex jamesii* Schw. Ann. Lyc. N. Y. 1:67. 1824.

James' sedge—dry or low, moist woodlands—southern Ontario to Michigan and Iowa, south to Maryland, Tennessee, Missouri, and Kansas. IOWA DISTRIBUTION: map 8-f. UNCONFIRMED COUNTY REPORTS: Henry (103) and Johnson (149).

27. *Carex eburnea* Boott in Hook. Fl. Bor. Amer. 2: 226. 1839.

*C. setifolia* Britt. in Britt. & Brown, Ill. Fl. 1: 332. 1896; not of Heer. 1840; nor of Kuntze 1849; nor of Godr. 1854 (B, BB).

Bristle-leaved sedge—rocky wooded slopes and ledges, sometimes on dry prairie—Newfoundland to Keewatin and Mackenzie, south to Virginia, Tennessee, Missouri, Nebraska, and British Columbia.

IOWA DISTRIBUTION: map 8-i. UNCONFIRMED COUNTY REPORTS: Dickinson (157) and Hardin (148).

28. *Carex richardsonii* R. Br. in Richards. in Frankl. Journey 751. 1823.

Richardson's sedge—dry open rocky places—western Ontario to Saskatchewan and Alberta, south to Indiana, Iowa, and South Dakota.

IOWA DISTRIBUTION: map 8-j. SPECIMENS EXAMINED: Iowa, without exact locality, 1876, Jones (G). Poweshiek County, Grinnell, June, 1879, Jones (ISC). UNCONFIRMED COUNTY REPORT: Plymouth (93).

29. *Carex pedunculata* Muhl. ex Willd. Sp. Pl. 4: 222. 1805.

Long-stalked sedge—dry woodlands, hillsides, and bluffs—Newfoundland to Ontario and Minnesota, south to Virginia, Pennsylvania, Michigan, Iowa, and South Dakota; also reported from British Columbia.

IOWA DISTRIBUTION: map 8-k.

30. *Carex limosa* L. Sp. Pl. 977. 1753.

Shore sedge; mud sedge—sphagnum bogs, hanging bogs, springy or muddy places along ponds or lakes—Newfoundland and Labrador to Yukon, south to Delaware, Ohio, Iowa, Montana, and northern California; also widely distributed in northern Eurasia.

IOWA DISTRIBUTION: map 8-j. SPECIMENS EXAMINED: Emmet County, 1878, Cratty (ISC), 1879, Cratty (ISC); Armstrong, June, 1883, Cratty (GRC, NY), June, 1884, Cratty (ISC), June, 1892, Cratty (SUI).

31. *Carex oligocarpa* Schkuhr ex Willd. Sp. Pl. 4: 279. 1805.

Few-fruited sedge—moist woodlands, frequently in dense shade—Massachusetts and Vermont to Ontario, Ohio, and Iowa, south to North Carolina, Alabama, and Texas.

IOWA DISTRIBUTION: map 8-l. UNCONFIRMED COUNTY REPORTS: Henry (103), Jasper (107), Johnson (149), Lee (92), and Muscatine (99).

32. *Carex hitchcockiana* Dewey, Amer. Jour. Sci. 10: 274. 1826.

Hitchcock's sedge—moist wooded slopes and floodplains—Massachusetts and Vermont to Ontario, Wisconsin, and Iowa, south to Virginia, Kentucky, and Missouri.

IOWA DISTRIBUTION: map 8-m. UNCONFIRMED COUNTY REPORT: Emmet (107). NOTES: (a) A report of this species from Johnson County (107) is based on a misidentification of a specimen of *C. hirtifolia*; a report from Lee County (107) is based on a misidentification of a specimen of *C. granularis*. (b) This species perhaps should not be maintained as distinct from *C. oligocarpa*; the distributional range of the two species are very

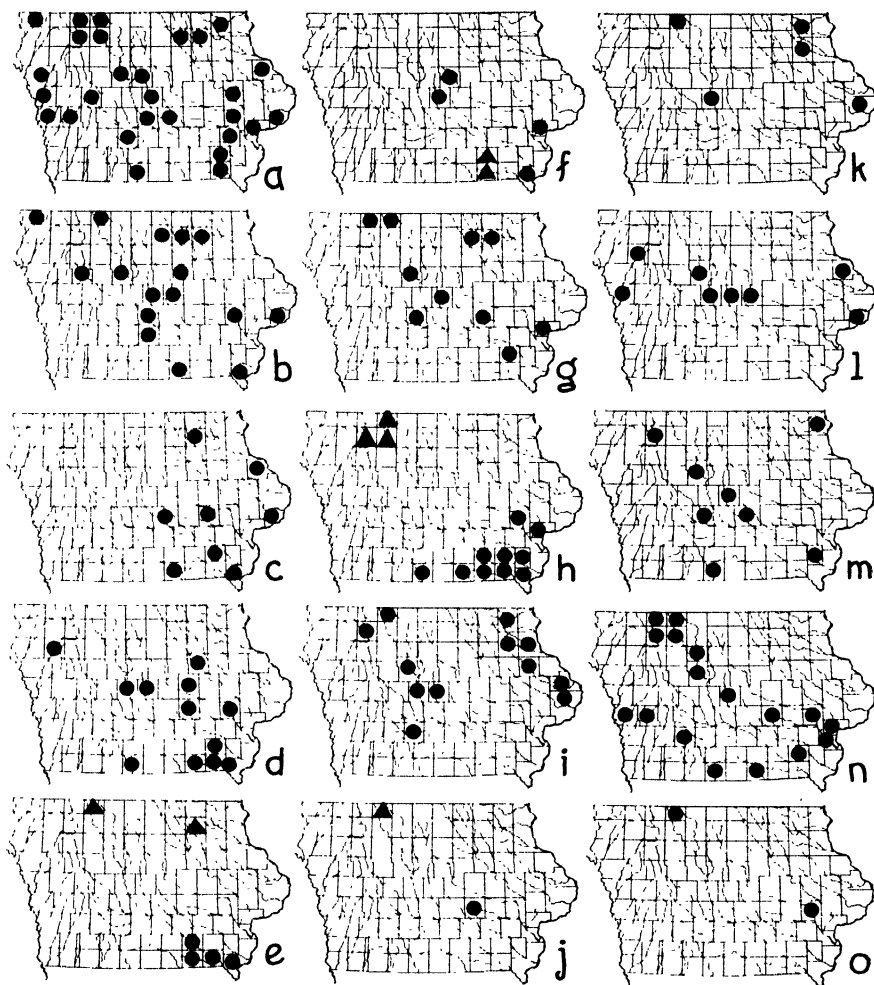


FIGURE 8. Distribution Maps of Iowa Cyperaceae.

- a—*Carex brevior*.
- b—*Carex bicknellii*.
- c—*Carex typhina*.
- d—*Carex davisii*.
- e—*Carex hirsutella* (•) and *Carex gracillima* (▲).
- f—*Carex bushii* (▲) and *Carex jamesii* (•).
- g—*Carex buxbaumii*.
- h—*Carex shortiana* (•) and *Carex tetanica* (▲).
- i—*Carex eburnea*.
- j—*Carex richardsonii* (•) and *Carex limosa* (▲).
- k—*Carex pedunculata*.
- l—*Carex oligocarpa*.
- m—*Carex hitchcockiana*.
- n—*Carex meadii*.
- o—*Carex crawei*.



much the same and both have essentially the same habitat preference. Additional field study will be required to settle this point.

33. *Carex tetanica* Schkuhr, Riedgr. Nachtr. 68. 1806.

*C. meadii* var. *bebbii* Arthur, Proc. Davenport Acad. 4:70. 1884.

Wood's sedge—wet prairie swales, sloughs, pond-edges, meadows, and low damp woodlands—Massachusetts to Ontario, Minnesota, and Alberta, south to New Jersey, Ohio, Iowa, and South Dakota.

IOWA DISTRIBUTION: map 8-g. NOTE: In the synonymy for this species, Mackenzie (53; page 238) includes the following citation: "*C. panicea Bebbii* Olney, Caric. Bor.-Am. 2 (name only); Arthur, Proc. Davenport Acad. 4:70. 1884." The combination, however, made by Arthur is not as cited by Mackenzie but as given in synonymy just above.

34. *Carex meadii* Dewey, Amer. Jour. Sci. 43:90. 1842.

"*C. tetanica*" of many Iowa authors; not of Schkuhr 1806.

*C. tetanica* var. *meadii* (Dewey) L. H. Bailey, Proc. Amer. Acad. 22:118. 1886 (G).

Mead's sedge—low or upland prairies and meadows—western New Jersey to Michigan, Minnesota, and Manitoba, south to Georgia, Tennessee, Arkansas, and Texas.

IOWA DISTRIBUTION: map 8-n. UNCONFIRMED COUNTY REPORTS: Boone (115), Hancock (107), Hardin (148), Lucas (107), and Scott (99, 107).

35. *Carex crawei* Dewey, Amer. Jour. Sci. II 2:246. 1846.

Crawe's sedge—dry prairies and meadows—Maine to Quebec, Manitoba, and Washington, south to northwestern New Jersey, New York, Ohio, Tennessee, Missouri, Wyoming, and Montana; also reported from southern Alabama.

IOWA DISTRIBUTION: map 8-o. SPECIMENS EXAMINED: Emmet County, without date, *Cratty* (G), *Paige* (ISC); Armstrong, June 15, 1884, *Cratty* (GRC, ISC, NY, SUI, W), June 1885, *Cratty* (ISC), June 9, 1904, *Cratty* (ISC). Johnson County, Iowa City, without date, *Hitchcock* (ISC). UNCONFIRMED COUNTY REPORT: Boone (115). NOTE: Reports of this species from Shelby County (107, 119) are based on misidentifications of a specimen of *C. meadii*.

36. *Carex granularis* Muhl. ex Willd. Sp. Pl. 4:279. 1805.

*C. haleana* Olney, Caric. Bor.-Amer. 6. 1871.

*C. granularis* var. *haleana* Porter, Proc. Acad. Philad. 1887:74. 1887.

*C. shriveri* Britt. Man. 208. 1901.

Meadow sedge—moist shaded woodlands, wet meadows, and wet cliffs—Maine to Quebec and Saskatchewan, south to Florida, Mississippi, Arkansas, and Kansas.

IOWA DISTRIBUTION: map 9-a. SPECIMENS EXAMINED: Clinton County, June 10, 1878, *Butler* 66 (ISC). Johnson County, June 1884, *Shimek* (ISC, SUI). Lee County, July 16, 1931, *Fults* 1432 (ISC); Keokuk, July

5, 1895, *Shimek* (SUI). Webster County, Fort Dodge, June 17, 1906, *Oleson* (ISC). UNCONFIRMED COUNTY REPORT: Henry (103).

37. *Carex conoidea* Schkuhr ex Willd. Sp. Pl. 4:280. 1805.

Field sedge—meadows and moist woodland—Newfoundland, southern Ontario, and Minnesota, south to Delaware, Pennsylvania, Ohio, and Iowa; reported from the mountains of North Carolina.

IOWA DISTRIBUTION: map 9-a. SPECIMEN EXAMINED: Fayette County, June 7, 1898, *Gardiner* (ISC). UNCONFIRMED COUNTY REPORTS: Floyd (107) and Scott (99, 107).

38. *Carex amphibola* Steud., var. *turgida* Fern. Rhodora 44:311. 1942.  
 "C. grisea" of many American authors; probably not of Wahl. 1803 (B, BB, BB2, G, R).

"C. amphibola" of some American authors; not of Steud. 1855.

"C. grisea var. *angustifolia*" of Iowa authors; not of Boott 1858.

Turgid sedge—dry or moist slopes, woodlands, and thickets—western New Brunswick to southern Ontario and Minnesota, south to Georgia, Alabama, Louisiana, and eastern Texas.

IOWA DISTRIBUTION: map 9-b. UNCONFIRMED COUNTY REPORTS: Allamakee (126), Boone (115), Chickasaw (126), Floyd (107), and Mahaska (96).

NOTES: (a) The reports of this species for Dubuque County (126, 145) are based on misidentifications of a specimen of *C. oligocarpa*. (b) Fernald (32) has presented evidence which seems to indicate that *C. grisea* Wahl. is really referable to *C. laxiculmis* Schw. or some other closely related species. (c) The typical phase of the species ranges from Virginia to Tennessee and Arkansas, south to Florida, Louisiana, and Texas, and another variety (the var. *rigida* Fern.) has a distribution much like that of var. *turgida*, except that it does not range quite so far westward.

39. *Carex laxiculmis* Schw. Ann. Lyc. N. Y. 1:70. 1824.

*C. digitalis* var. *copulata* L. H. Bailey, Mem. Torrey Club 1:47. 1889.

*C. laxiculmis* var. *copulata* (L. H. Bailey) Fern. Rhodora 8:183. 1906.

*C. copulata* (L. H. Bailey) Mackenz. N. Amer. Fl. 18:251. 1935.

Spreading sedge—dry woodlands, thickets, and wooded ravines—Maine to southern Ontario, Wisconsin, and Iowa, south to North Carolina, West Virginia, Ohio, and Missouri.

IOWA DISTRIBUTION: map 9-c. SPECIMENS EXAMINED: Black Hawk County, May, 1929, *Burk* 276 (T). Muscatine County, Wildcat Den State Park, June, 1897, *Barnes and Miller* (ISC). NOTES: (a) The report of *C. digitalis* for Johnson County (164) undoubtedly should be referred to this species. (b) The report of this species for Hardin County (148) was based on a misidentification of a specimen of *C. alburna*. (c) Fernald (32) has recently stated the belief that most of the specimens that have been identified as *C. copulata*, including the type of that species, represent a series of hybrids between *C. laxiculmis* and *C. digitalis* Willd.

40. *Carex blanda* Dewey, Amer. Jour. Sci. 10:45. 1825.

"*C. laxiflora*" of numerous Iowa authors; not of Lam. 1791.

*C. laxiflora* var. *striatula* Carey in A. Gray, Man. ed. 2. 524, in part. 1856.

*C. laxiflora* var. *blanda* (Dewey) Boott, Ill. Carex 37. 1858 (B, BB, G).

*C. laxiflora* var. *varians* L. H. Bailey, Mem. Torrey Club 1:32, in part. 1889.

Woodland sedge—moist wooded slopes, dry and open woodlands—New Hampshire and Quebec to Minnesota and North Dakota, south to Georgia, Alabama, Louisiana, and Texas.

IOWA DISTRIBUTION: map 9-d. UNCONFIRMED COUNTY REPORTS: Dickinson (157), Floyd (107), Jasper (107), Mahaska (96), Muscatine (99, 107), Scott (99, 107), Shelby (119), and Winnebago (107). NOTES: (a) The report of *C. laxiflora* from Mahaska County (96) probably should be referred to this species. (b) This is an extremely variable species; some Iowa specimens approximate *C. gracilescens* Steud. (*C. laxiflora* var. *gracillima* (Boott) Robins. & Fern.), others are typical of *C. blanda*, and still others tend toward *C. albursina*; considerable field work is needed to determine the extent of and exact limits of variability of the species in this group (the Section *Laxiflorae* of Mackenzie's treatment of the genus).

41. *Carex albursina* Sheldon, Bul. Torrey Club 20:284. 1893.

*C. laxiflora* var. *latifolia* Boott, Ill. Carex 38. 1852 (G).

White bear sedge—moist woodlands and gullies—Connecticut, Vermont, and Quebec to Minnesota, south to Virginia, Tennessee, and Arkansas.

IOWA DISTRIBUTION: map 9-e. UNCONFIRMED COUNTY REPORTS: Dickinson (157), Henry (103), Linn (133), and Scott (99, 107).

42. *Carex assiniboinensis* W. Boott, Bot. Gaz. 9:91. 1884.

Assiniboia sedge—moist woodlands—northern Wisconsin to Manitoba, south to northern Iowa and South Dakota.

IOWA DISTRIBUTION: map 9-c. SPECIMENS EXAMINED: Emmet County, Mud Lake, July 20, 1916, *Wolden* (G, ISC), June 13, 1922, *Wolden* 447 (ISC), June 19, 1923, *Wolden* 814a (W); Wallingford, 1911, *Wolden* (ISC). Hardin County, Iowa Falls, without date, *Peck* (ISC). Winneshiek County, Decorah Township, June 2, 1934, *Tolstead* (ISC). UNCONFIRMED COUNTY REPORT: Boone (115). NOTE: Tolstead (166a) has just reported an interesting series of observations on stoloniferous growth in this species.

43. *Carex hirtifolia* Mackenz. Bul. Torrey Club 37:244. 1910.

*C. pubescens* Muhl. ex Willd. Sp. Pl. 4:281. 1805; not of Poir. 1789; nor of Gilib. 1792 (B, BB, G).

Hairy sedge—dry woodlands and thickets—New Brunswick and Quebec to Minnesota, south to Maryland, Kentucky, Missouri and eastern Kansas.

IOWA DISTRIBUTION: map 9-f. UNCONFIRMED COUNTY REPORTS: Decatur (87), Dickinson (157), Hardin (148), Henry (103), Jasper (107), Muscatine (99, 107), and Scott (99, 107).

## KEY TO IOWA CAREX—GROUP E

- A. Pistillate scales obtuse, acute, or mucronate at apices; spikes sessile and more or less aggregated on stiff peduncles.
- B. Beaks of perigynia formed by the gradual tapering of the bodies, serrulate margined, obliquely-cut (thus more or less bidentulate in age) at apices, not less than one-fourth the length of the perigynium bodies.
- C. Perigynia prominently nerved on both faces .....44. *C. chordorrhiza*
- CC. Perigynia nerveless or obscurely nerved at base on ventral surface, more or less nerved or nerveless dorsally.
- D. Plants forming colonies, spreading by long-creeping rhizomes, the culms arising singly or in small clumps at intervals.
- E. Ventral surface of leaf-sheaths green-striate (similar, in texture and color, to the dorsal surface) to the base of the blade, the sheath prolonged upward into a conspicuous hyaline tubular ligule. ....45. *C. sartwellii*
- EE. Ventral surface of leaf-sheaths completely hyaline, truncate at base of blade; ligule inconspicuous.
- F. Beaks of perigynia about one-half as long as bodies; spikes few and aggregated, or numerous and more or less separated; individual plants monoecious (bearing both staminate and pistillate flowers).
- G. Culms obtusely angled, smooth; leaves 1.5 mm. or less in width at base, narrower above, stiff, more or less involute; pistillate scales rounded or obtuse at apices, broadest above the middle .....46. *C. eleocharis*
- GG. Culms sharply triangular, usually roughened on the angles above; leaves 1.5 mm.-3 mm. wide, firm but not stiff, flattened or somewhat channeled above but not involute; pistillate scales tapering to apices, acute or cuspidate, broadest at or near the base .....47. *C. praegracilis*
- FF. Beaks of perigynia as long as the bodies; spikes numerous, aggregated into a dense heavy head; individual plants usually dioecious (bearing only staminate or only pistillate flowers) or rarely monoecious .....48. *C. douglasii*
- DD. Plants cespitose, forming large dense clumps; roots fibrous; rhizomes, if present, very short.
- E. Ventral surface of leaf-sheaths white-hyaline; spikes usually closely aggregated; perigynia shining .....49. *C. diandra*
- EE. Ventral surface of leaf-sheaths copper-colored, at least at the mouth; lower spikes more or less separated, the inflorescence thus interrupted; perigynia dull .....50. *C. prarisa*
- BB. Beaks of perigynia formed by the more or less abrupt contraction of the bodies, entire at apex, minute (not more than one-tenth the length of the body) or the perigynia beakless.
- C. Perigynia ovate or elliptical, broadest at the middle or base; pistillate scales ovate, acute at the apices; plants, including culm-bases, slender.
- D. Perigynia elliptical, broadest at the middle, narrowed toward both base and apex, brown or green when mature.
- E. Perigynia more or less flattened, not inflated at apices, greenish or dark green at maturity, punctulate but not granular roughened; leaf-sheaths sometimes minutely pubescent dorsally .....51. *C. stricta*
- EE. Perigynia inflated at apex, brown at maturity, more or less granular roughened; leaf-sheaths always glabrous dorsally .....52. *C. haydenii*
- DD. Perigynia ovate, broadest at the base, slightly constricted at the middle, straw-colored at maturity .....53. *C. emoryi*
- CC. Perigynia obovate, broadest at the apices; pistillate scales spatulate or obovate, obtuse at the apices; plants usually with stout, more or less spongy (at least at the base) culms .....54. *C. aquatilis*  
var. *altior*
- AA. Pistillate scales more or less retuse at apices, the midribs prolonged as ciliate-scabrid awns which much exceed the perigynia in length; spikes lax and drooping on slender peduncles .....55. *C. crinita*

44. *Carex chordorrhiza* L. f. Suppl. 414. 1781.

Creeping sedge—sphagnum bogs and lake shores—Newfoundland and Labrador to Keewatin, south to New York, Ohio, Iowa, and Saskatchewan. IOWA DISTRIBUTION: map 9-g. SPECIMENS EXAMINED: Emmet County, without date, *Cratty* (ISC), May 1878, *Cratty* (SUI), 1881, *Cratty* (G), 1884, *Cratty* (G, ISC).

45. *Carex sartwellii* Dewey, Amer. Jour. Sci. 43:90. 1842.

"*C. disticha*" of some American authors; not of Huds. 1762.

Sartwell's sedge—marshes, meadows, moist prairies, edges of bogs, and lake margins, sometimes in water up to 18 inches deep—western New York and Ontario to Saskatchewan and British Columbia, south to Ohio, Missouri, Nebraska, Colorado, and Montana.

IOWA DISTRIBUTION: map 9-h. UNCONFIRMED COUNTY REPORTS: Black Hawk (104), Dickinson (89, 157), Hardin (148), and Muscatine (99).

46. *Carex eleocharis* L. H. Bailey, Mem. Torrey Club 1:6. 1889.

"*C. stenophylla*" of many American authors; not of Wahl. 1839 (B, BB, BB2, G).

Spike-rush sedge; involute-leaved sedge—prairies, plains, and open gravelly ridges; occasionally in dry open woodlands—Manitoba to Yukon, south to northwestern Iowa, Kansas, New Mexico, Utah, and eastern Oregon.

IOWA DISTRIBUTION: map 9-i. UNCONFIRMED COUNTY REPORT: Winnebago (98).

47. *Carex praeegracilis* W. Boott, Bot. Gaz. 9:87. 1884.

*C. camporum* Mackenz. Bul. Torrey Club 27:244. 1910 (BB2).

Clustered field sedge—moist spots and swales in prairies and plains and in the foothills—Manitoba to Yukon and British Columbia, south to Iowa, Oklahoma, Arizona, northern Mexico, and California; also reported from Michigan.

IOWA DISTRIBUTION: map 9-j. UNCONFIRMED COUNTY REPORT: Palo Alto (128).

48. *Carex douglasii* Boott in Hook. Fl. Bor. Amer. 2:213. 1839.

Douglas' sedge—dry open prairies and plains, in sandy or alkaline soil—Manitoba to British Columbia, south to Nebraska, Colorado, New Mexico, and California; also in eastern Iowa.

IOWA DISTRIBUTION: map 9-g. SPECIMEN EXAMINED: Jasper County, Sugar Creek, May 27, 1904 (or 1905), *Rosa Drew* (GRC); all flowers on this specimen are pistillate. NOTE: This is the first report of this western species for Iowa. The nearest localities from which the species has been reported previously—Leeds, North Dakota, Eagle's Nest Butte, South Dakota, and the sand hills of central Nebraska—are between 385 and 560 miles distant from the single Iowa locality.

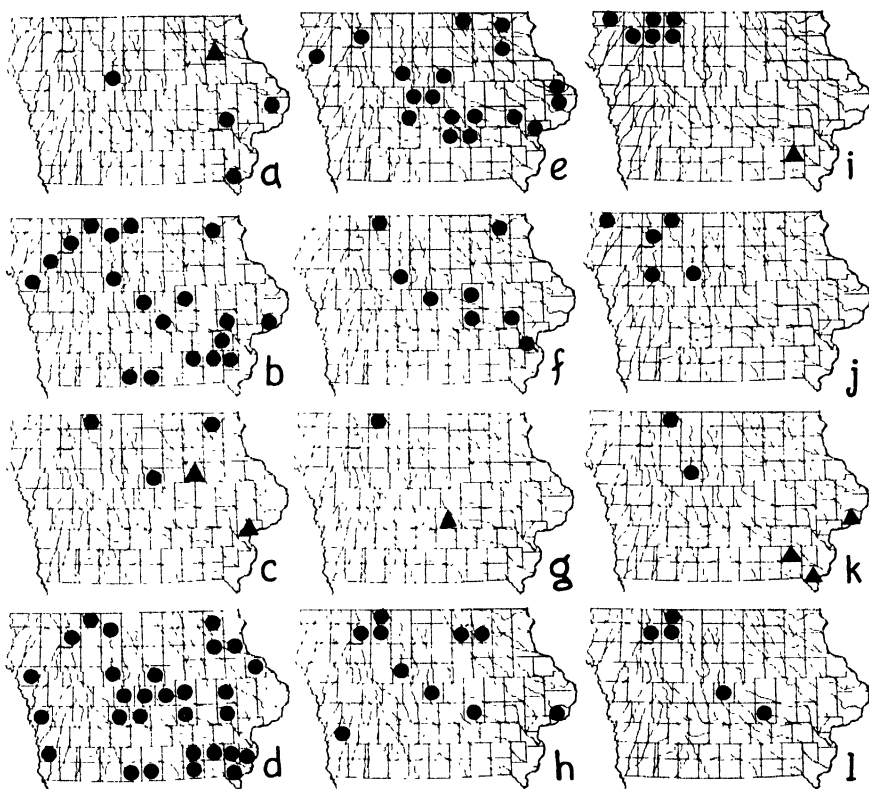


FIGURE 9. Distribution Maps of Iowa Cyperaceae.

- a—*Carex granularis* (●) and *Carex conoidea* (▲).
- b—*Carex amphibola* var. *turgida*.
- c—*Carex laxiculmis* (▲) and *Carex assiniboinensis*. (●).
- d—*Carex blanda*.
- e—*Carex albursina*.
- f—*Carex hirtifolia*.
- g—*Carex chordorrhiza* (●) and *Carex douglasii* (▲).
- h—*Carex sartwellii*.
- i—*Carex eleocharis* (●) and *Carex crinita* (▲).
- j—*Carex praegracilis*.
- k—*Carex diandra* (●) and *Carex leavenworthii* (▲).
- l—*Carex prarisa*.

49. *Carex diandra* Schrank, Cent. Bot. Anmerk. 57. 1781.

*C. teretiuscula* Gooden. Trans. Linn. Soc. 2:163. 1794 (BB).

Lesser panicked sedge—wet meadows and prairie swales—Newfoundland and Labrador to Yukon, south to New Jersey, Indiana, Nebraska, Colorado, and California.

IOWA DISTRIBUTION: map 9-k. SPECIMENS EXAMINED: Emmet County, June, 1878, *Cratty* (ISC), 1883, *Cratty* (ISC); Armstrong, July 3, 1904, *Cratty* (NY); Emmet Township, July 15, 1925, *Wolden 1102a* (W), June 16, 1928, *Wolden 1357* (W). Webster County, Fort Dodge, without date, *Paige* (ISC). UNCONFIRMED COUNTY REPORTS: Hardin (148), Poweshiek (93, 107), and Story (107).

50. *Carex prarisa* Dewey in Wood, Class-Book 414. 1845.

*C. prairea* Dewey in Wood, Class-Book, ed. 2. 578 (*sphalm?*). 1847 (BB2).

*C. teretiuscula* var. *ramosa* Boott, Ill. Carex 145. 1867.

*C. teretiuscula* var. *prairea* (Dewey) Britt. in Britt. and Brown, Ill. Fl. 1:344. 1896 (B, BB).

*C. diandra* var. *ramosa* (Boott) Fern. Rhodora 10:48. 1908 (G).

Prairie sedge—wet meadows, wet prairies, bogs, and around cold hillside springs—Vermont and Quebec to Saskatchewan, south to New Jersey, Ohio, Iowa, and Nebraska.

IOWA DISTRIBUTION: map 9-l. UNCONFIRMED COUNTY REPORT: Lyon (107). NOTES: (a) A report of this species from Story County (107) is based on a misidentification of a specimen of *C. sartwellii*. (b) There is no evidence available to show that "*prairea*" was intended as a correction for "*prarisa*"; as attempts at associating this species with its prairie habitat both are equally bad Latin. The epithet "*prairea*" should be considered as either an error or as a superfluous name (Art. 60 of the *International Rules of Botanical Nomenclature*); it is unfortunate that this epithet has been perpetuated by so many authors.

51. *Carex stricta* Lam. Encyc. 3:367. 1791.

*C. strictior* Dewey in Wood, Class-Book 582. 1845.

*C. stricta* var. *strictior* (Dewey) Carey in A. Gray, Man. 548. 1848.

*C. stricta* var. *angustata* L. H. Bailey in A. Gray, Man. ed. 6. 600. 1890.

Tussock sedge—swampy woodlands, wet meadows, low wet prairie, prairie sloughs, hanging bogs, and shallow water along lake shores—Nova Scotia to Quebec and Minnesota, south to North Carolina, Ohio, and Iowa; also reported from Texas.

IOWA DISTRIBUTION: map 10-a. UNCONFIRMED COUNTY REPORTS: Boone (141), Chickasaw (126), Decatur (87, 107), Dickinson (107), Hamilton (142), Hardin (148), Henry (103), Linn (107), Mahaska (96), Muscatine (99), Pottawattamie (124), and Shelby (118). NOTES: (a) A report of this species from Poweshiek County (107) is based on a misidentification of a specimen of *C. emoryi*. (b) As in Indiana, where Hermann (44) reports considerable intergradation and extreme inconstancy of characters

for the complex, I find the Iowa material difficult to separate into two entities; and when the specimens from the total range of both "species" are taken into consideration the line of demarcation between them becomes even less tangible. For these reasons, I do not accept the "*strictior*" phase of the species—which in the extreme variant is a non-cespitose plant which forms colonies instead of tussocks and has pistillate scales longer than the perigynia and the ventral surface of the leaf sheaths finely pubescent—as constituting even a variety.

52. *Carex haydenii* Dewey, Amer. Jour. Sci. II 18:103. 1854.

"*C. aperta*" of some authors; not of Boott. 1839.

*C. stricta* var. *decora* L. H. Bailey, Bot. Gaz. 13:85. 1888 (G).

Hayden's sedge—swampy meadows, marshes, prairie sloughs, and wet roadside ditches—New Brunswick to Ontario and Minnesota, south to New Jersey, Pennsylvania, Indiana, Missouri, and Nebraska.

IOWA DISTRIBUTION: map 10-b. UNCONFIRMED COUNTY REPORTS: Muscatine (99, 107) and Scott (99, 107).

53. *Carex emoryi* Dewey in Torr. Bot. Mex. Bound. Survey 230. 1859.

Emory's sedge—swampy meadows, wet prairies, marshes, river banks, and along ponds and lakes—western New Jersey to Michigan, Minnesota, and Manitoba, south to Virginia, Indiana, Missouri, Oklahoma, Texas, and New Mexico.

IOWA DISTRIBUTION: map 10-c. UNCONFIRMED COUNTY REPORT: Winneshiek (124).

54. *Carex aquatilis* Wahl., var. *altior* (Rydb.) Fern. Rhodora 44:295. 1942.

"*C. aquatilis*" of some American authors; not of Wahl. 1803 (B, BB, BB2, G, R).

*C. variabilis* var. *altior* Rydb. Mem. N. Y. Bot. Gard. 1:76. 1900.

*C. aquatilis* var. *substricta* Kük. Pflanzenr. 38[IV. 20]:309. 1909.

*C. substricta* (Kük.) Mackenz. in Rydb. Fl. Rocky Mts. 139. 1932.

Water sedge—sloughs, marshes, swamps, wet meadows, and prairies, hanging bogs, lake and pond edges, sometimes in water as much as two feet deep—Newfoundland to Quebec, Manitoba, Saskatchewan, and British Columbia, south to New Jersey, Indiana, Missouri, Nebraska, Colorado, and Washington.

IOWA DISTRIBUTION: map 10-d. UNCONFIRMED COUNTY REPORT: Pottawatamie (124). NOTES: (a) The reports of this species from Fayette County (107, 117) are based on misidentifications of specimens of *C. stricta*. (b) Fernald (32) recognizes the American material of this species as being varietally distinct from the typical phase of the species which is widely distributed through northern Eurasia, in the arctic and sub-arctic regions of North America, and in the higher western mountains as far south as New Mexico and California.



55. *Carex crinita* Lam. Encyc. 3: 393. 1791.

Fringed sedge—wet woodland margins and thickets, in swamps, swales, and prairie sloughs—Nova Scotia and Quebec to Minnesota, south to North Carolina, Tennessee, Arkansas, Louisiana, and Texas.

IOWA DISTRIBUTION: map 9-i. SPECIMENS EXAMINED: Jefferson County, Cedar Township, July 10, 1933, *McDonald* 807 (P), May 30, 1934, *Gilly and McDonald* 1821 (P). UNCONFIRMED COUNTY REPORT: Story (124).

NOTE: The terminal spikes of this species are normally staminate; occasionally, however, the terminal spike is either gynecandrous, or androgynous, or sometime with pistillate flowers in the middle and with staminate flowers at both apex and base.

## KEY TO IOWA CAREX—GROUP F

- A. Perigynia scarcely (if at all) basally spongy-thickened, the achenes thus almost entirely filling the bodies of the perigynia.
  - B. Spikes closely aggregated into a head, the individual spikes varying from globose to scarcely more than twice as long as thick.
    - C. Perigynia truncate-cordate and distinctly broadest at base ..... 56. *C. leavenworthii*
  - CC. Perigynia rounded or tapering toward the base, broadest somewhat above the base or at the middle.
    - D. Bodies of the pistillate scales almost as long as the perigynia.
      - E. Dorsal surfaces of leaf-sheaths entirely green, firm-textured; perigynia conspicuously nerved dorsally, nerved or nerveless ventrally ..... 57. *C. muhlenbergii*
      - EE. Dorsal surfaces of leaf-sheaths green and white mottled or white-hyaline between the green ribs; perigynia obscurely nerved or nerveless dorsally, nerveless ventrally ..... 58. *C. grvida*
    - DD. Bodies of pistillate scales only half as long as perigynia.
      - E. Apices of pistillate scales acute, the midribs prolonged as definite mucros ..... 59. *C. cephalophora*
      - EE. Apices of pistillate scales obtuse or acutish, the midribs not mucronate ..... 60. *C. cephaloidea*
  - BB. Spikes, especially the lower, more or less widely separated, the individual spikes normally oblong-cylindrical, twice as long or more than twice as long as thick.
    - C. Leaves 5 mm.-10 mm. wide; lowermost spikes simple, solitary; leaf-sheaths (except rarely the lowermost) not cross-rugulose ventrally; pistillate scales acute or mucronate at apices ..... 61. *C. sparganioides*
    - CC. Leaves 5 mm. or less in width; lowermost spikes compound or clustered; leaf-sheaths strongly cross-rugulose ventrally; pistillate scales with midribs prolonged as scabrid awns.
      - D. Beaks of the perigynia shorter than the bodies, the bodies 1.5 mm.-2 mm. wide; culms normally longer than the leaves ..... 62. *C. annectens* var. *xanthocarpa*
      - DD. Beaks of the perigynia as long as the bodies, the bodies 1 mm.-1.5 mm. wide; culms normally shorter than the leaves ..... 63. *C. vulpinoidea*
  - AA. Perigynia more or less spongy-thickened or swollen basally, the achenes thus filling only the upper portion of the perigynia.
    - B. Spikes few-flowered, distant, the perigynia radiating in all directions; culms slender and weak; leaves thin, flaccid, 1 mm.-2.5 mm. (or rarely 3 mm.) wide.
      - C. Stigmas long, slender, usually straight, light red; perigynia gradually tapering into beak, usually 3 mm.-3.5 mm. long; spikes with 6-12 perigynia; leaves with blades 1 mm.-2 mm. wide ..... 64. *C. rosea*

- CC. Stigmas short, stout, usually twisted, dark red; perigynia abruptly contracted into beak, usually 3.25 mm.-4.5 mm. long; spikes with 6-20 perigynia; leaf blades 1.5 mm.-4 mm. (averaging about 2.5 mm.) wide ..... 64a. *C. rosea* var. *pusilla*
- BB. Spikes either few-flowered and aggregated into a terminal head, or numerous-flowered and more or less separated; perigynia appressed-ascending or spreading; culms stout, firm; leaves firm, 3 mm. or more in width.
- C. Basal spongy-thickened portion of perigynia merely truncate-rounded; beaks of the perigynia gradually tapering, less than twice as long as bodies.
- D. Culms flattened, conspicuously winged; perigynia nerveless ventrally.
- E. Beaks of perigynia about the length of the bodies, the lanceolate bodies faintly nerved dorsally; ventral surface of leaf-sheaths hyaline and smooth ..... 65. *C. alopecoidea*
- EE. Beaks of perigynia half the length of the bodies, the broadly ovate bodies strongly nerved dorsally; ventral surface of leaf-sheaths hyaline and strongly cross-rugulose ..... 66. *C. conjuncta*
- DD. Culms triangular, scarcely (if at all) winged; perigynia conspicuously nerved ventrally; ventral surface of leaf-sheaths either smooth or cross-rugulose.
- E. Ventral surface of leaf-sheaths usually conspicuously cross-rugulose and prolonged upwards at mouth beyond base of leaf blade; pistillate scales merely acute or mucronulate ..... 67. *C. stipata*
- EE. Ventral surface of leaf sheaths smooth, concave at mouth and not prolonged upwards beyond base of leaf blade; pistillate scales conspicuously awned ..... 67a. *C. stipata*  
var. *laevivaginata*
- CC. Basal one-third of each perigynium swollen and disk-like; beaks of perigynia slender, two or three times as long as bodies. .... 68. *C. crus-cervi*

56. *Carex leavenworthii* Dewey, Amer. Jour. Sci. II 2:246. 1846.

Leavenworth's sedge—dry prairies and woodlands—southern New Jersey and eastern Pennsylvania, south along coastal plain to Florida and west to Texas; northward in the Mississippi Valley to Indiana, Illinois, and Iowa; also reported from southwestern Ontario.

IOWA DISTRIBUTION: map 9-k. SPECIMENS EXAMINED: Jefferson County, Center Township, May 27, 1933, *Gilly and McDonald* 369, in part (P). Lee County, Keokuk, June 1, 1897, *Shimek* (ISC, SUI). Scott County, Buffalo, June, 1895, *Barnes and Miller* (ISC). UNCONFIRMED COUNTY REPORTS: Henry (103), Johnson (164), and Shelby (107).

57. *Carex muhlenbergii* Schkuhr, Riedgr. Nachtr. 12. 1806.

*C. muhlenbergii* var. *enervis* Boott, Ill. Carex 124. 1862.

*C. plana* Mackenz. Bul. Torrey Club 50:350. 1923.

Muhlenberg's sedge—dry hillsides, frequently in sandy soil—Maine to Ontario, Minnesota, and Nebraska, south to Florida, Louisiana, and Texas; most common along the coastal plain and in the Mississippi Valley.

IOWA DISTRIBUTION: map 10e. UNCONFIRMED COUNTY REPORT: Muscatine (164). NOTE: Although some of the Iowa material appears to be referable, when examined casually, to *C. plana*, I am unable—on the basis of the examination of a large series of specimens, including those examined by Mackenzie—to satisfactorily separate that "species" from *C. muhlenbergii*, even as a variety.

58. *Carex gravida* L. H. Bailey, Mem. Torrey Club 1:5. 1889.*C. gravida* var. *larifolia* L. H. Bailey, l. c., 6.*C. agglomerata* Mackenz. Bul. Torrey Club 33:442. 1906; not of C. B. Clarke 1903.*C. aggregata* Mackenz. Bul. Torrey Club 37:246. 1910.*C. lunelliana* Mackenz. Bul. Torrey Club 42:615. 1915.*C. gravida* var. *lunelliana* (Mackenz.) Hermann, Amer. Midl. Nat. 17:855. 1936.

Heavy sedge—open woods, dry hillsides, prairie sloughs and swales, frequently along roadsides—western New Jersey and southern Ontario to Minnesota, North Dakota, and Wyoming, south to Virginia, Kentucky, Arkansas, Texas, and New Mexico.

IOWA DISTRIBUTION: map 10-f. UNCONFIRMED COUNTY REPORTS: Allamakee (126), Marshall (107), Mahaska (96), Muscatine (99, 107, 156, 159), Poweshiek (107), and Van Buren (107). NOTE: I am unable to satisfactorily separate, even as varieties, the several "species" whose names are included in synonymy above. Individual specimens, it is true, are usually clearly referable to one or the other of them, but when the available material of the complex is considered no really limiting characters or groups of characters can be isolated.

59. *Carex cephalophora* Muhl. ex Willd. Sp. Pl. 4:220. 1805.

Oval-headed sedge—dry open woodlands—Maine and western Quebec to Manitoba and South Dakota, south to Florida, Louisiana, and Texas.

IOWA DISTRIBUTION: map 10-g. UNCONFIRMED COUNTY REPORTS: Allamakee (126, 160), Decatur (87), Fayette (107, 117), Louisa (102, 107), Lyon (151, 153), Muscatine (99, 102, 107, 156, 159), Pottawattamie (120), and Scott (107).

60. *Carex cephaloidea* Dewey, Rep. Pl. Mass. 262. 1840.

Thin-leaved sedge—low woodlands—New Brunswick to Ontario and Minnesota, south to New Jersey, Ohio, and Iowa.

IOWA DISTRIBUTION: map 10-h. UNCONFIRMED COUNTY REPORT: Henry (103). NOTE: The reports of this species from Scott County (99, 107) are based on misidentifications of specimens of *C. gravida*.

61. *Carex sparganioides* Muhl. ex. Willd. Sp. Pl. 4:237. 1805.

Bur-reed sedge—wooded slopes and ravines and floodplain thickets—New Hampshire to Quebec, Minnesota, and South Dakota, south to Virginia, Kentucky, Missouri, and Kansas.

IOWA DISTRIBUTION: map 10-i. UNCONFIRMED COUNTY REPORTS: Clayton (126), Dubuque (126), Fayette (126), Floyd (107), Hardin (148), Henry (103), Mahaska (96), Muscatine (99, 107), Scott (99, 107), and Winne-shiek (124, 155). NOTE: Reports of this species from Dallas County (107) and Emmet County (170) are based on misidentifications of *C. gravida*.

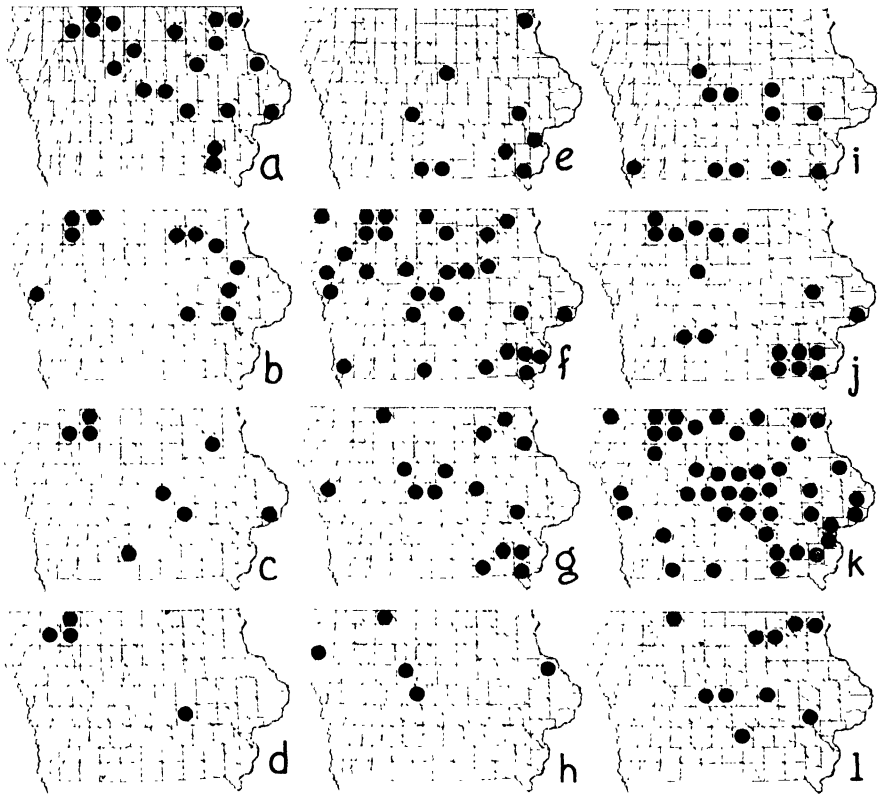


FIGURE 10. Distribution Maps of Iowa Cyperaceae.

- a—*Carex stricta*.
- b—*Carex haydenii*.
- c—*Carex emoryi*.
- d—*Carex aquatilis* var. *altior*.
- e—*Carex muhlenbergii*.
- f—*Carex gravida*.
- g—*Carex cephalophora*.
- h—*Carex cephaloidea*.
- i—*Carex sparganioides*.
- j—*Carex annectens* var. *xanthocarpa*.
- k—*Carex vulpinoidea*.
- l—*Carex rosea*.

62. *Carex annectens* (Bickn.) Bickn., var. *xanthocarpa* (Bickn.) Wiegand, *Rhodora* 24: 74. 1922.

*C. xanthocarpa* Bickn. *Bul. Torrey Club* 23: 22. 1896; not of Degl. 1807 (B).

"*C. xanthocarpa* var. *annectens*" of some authors; not of Bickn. 1896.

"*C. setacea* var. *ambigua*" of some Iowa authors; not of Fern. 1906 (G).

"*C. annectens*" of some Iowa authors; not of Bickn. 1908.

*C. brachyglossa* Mackenz. *Bul. Torrey Club* 50: 355. 1923.

Yellow-fruited sedge—fields, pastures, prairies, woodland edges, and pond banks; frequently becoming a weed—Maine to New York, Michigan, Wisconsin, and Iowa, south to Virginia, Indiana, Missouri, and Kansas.

IOWA DISTRIBUTION: map 10-j. UNCONFIRMED COUNTY REPORT: Hardin (148). NOTES: (a) The report of this species from Black Hawk County (104) was based on a misidentification of a specimen of *C. vulpinoidea*. (b) Although the larger fruited phase of the species (typical *annectens*) has been reported for Iowa, I find that the specimens on which such reports are based are quite characteristic of this variety; because of intergradation in many and extensive areas within the range of the species, I am unable to accept specific status for this variety.

63. *Carex vulpinoidea* Michx. *Fl. Bor.-Amer.* 2: 169. 1803.

*C. setacea* Dewey, *Amer. Jour. Sci.* 9: 61. 1825.

Fox sedge—low wet prairies, marshes, swamps, river floodplains, banks of ponds and streams, sometimes in shallow water—Newfoundland to Ontario, Manitoba, and British Columbia, south to Florida, Texas, Arizona, Idaho, and Oregon.

IOWA DISTRIBUTION: map 10-k. UNCONFIRMED COUNTY REPORTS: Appanoose (117), Benton (107), Cedar (107), Lee (121), Lucas (107), Page (120), and Wright (107).

64. *Carex rosea* Schkuhr ex. Willd. *Sp. Pl.* 4: 237. 1805.

"*C. rosea* var. *radiata*" of Iowa authors; not of Dewey 1826.

Stellate sedge—dry or moist woodlands and thickets—Nova Scotia and Quebec to Manitoba and North Dakota, south to Georgia, Louisiana, Arkansas, and Kansas.

IOWA DISTRIBUTION: map 10-l. UNCONFIRMED COUNTY REPORTS: Henry (103), Muscatine (107), Pottawattamie (120), Scott (99, 107), and Van Buren (107). NOTES: (a) These unconfirmed reports, or at least some of them, may refer to the var. *pusilla* rather than to the typical phase of the species. (b) Although this species-complex is usually treated as three or more separate species, I am unable to find sufficiently clear distinctions between such entities. In addition to the variety recognized just below, *C. radiata* (Wahl.) Dewey ex Chapm., *C. retroflexa* Muhl. and *C. texensis* (Torr.) L. H. Bailey—three phases of the species-complex which have not been collected in Iowa—should be treated as varieties instead of as species.

64a. *Carex rosea* Schkuhr ex Willd., var. *pusilla* Peck ex Howe, Ann. Rept. N. Y. State Mus. 48:132. 1897.

*C. convoluta* Mackenz. Bul. Torrey Club 43:428. 1916.

Coarse stellate sedge; twisted-beak sedge—dry woodlands—Nova Scotia and Quebec to Manitoba and South Dakota, south to South Carolina, Alabama, Tennessee, Arkansas, and Kansas.

IOWA DISTRIBUTION: map 11-a. NOTE: This variety is more widespread and of more common occurrence in the state than the typical phase of the species.

65. *Carex alopecoidea* Tuckerm. Enum. Caric. 18. 1843.

Foxtail sedge—low swampy meadows and moist woodlands—Maine and Quebec to Manitoba, south to New Jersey, Indiana, and Iowa.

IOWA DISTRIBUTION: map 11-b. SPECIMENS EXAMINED: Clay County, Gillett's Grove Township, June 27, 1936, *Hayden* 670 (ISC, NY); Hardland Township, June 28, 1936, *Hayden* 669 (ISC). Emmet County, Armstrong, June, 1904, *Cratty* (ISC), June 14, 1905, *Cratty* (ISC); Estherville, June 11, 1926, *Wolden* 1218 (NY), June 20, 1926, *Wolden* 1222 (ISC, W). Palo Alto County, Mud Lake, May 5, 1937, *Hayden* 8084 (ISC). UNCONFIRMED COUNTY REPORT: Story (92).

66. *Carex conjuncta* Boott, Ill. Carex 122. 1862.

Soft fox sedge—low wet woodlands—New Jersey and New York to Michigan, Illinois, Iowa, and South Dakota, south to Virginia, Kentucky, Missouri, and eastern Kansas.

IOWA DISTRIBUTION: map 11-c. UNCONFIRMED COUNTY REPORTS: Henry (103), Jasper (107), and Muscatine (162).

67. *Carex stipata* Muhl. ex Willd. Sp. Pl. 4:233. 1805.

Awl-fruited sedge—swamps, marshes, wet meadows, prairie swales, and sometimes in low open swampy woodlands—Newfoundland to Manitoba, British Columbia, and southern Alaska, south to North Carolina, Tennessee, Missouri, Kansas, New Mexico, and California.

IOWA DISTRIBUTION: map 11-d. UNCONFIRMED COUNTY REPORTS: Decatur (87), Dickinson (157), Muscatine (99, 107), Scott (99, 107), Shelby (107), and Story (107). NOTE: Some of these reports, particularly those from the southern part of the state, may refer to the following variety instead of to the typical form of the species.

67a. *Carex stipata* Muhl. ex Willd., var. *laevivaginata* Kük. Pflanzenr. 38[IV. 20]:172. 1909.

*C. laevivaginata* (Kük.) Mackenz. in Britt. & Brown, Ill. Fl. ed. 2. 1:371. 1913.

Smooth-sheath sedge—essentially the same habitat as the species—Massachusetts to New York, Michigan, and Minnesota, south to Florida, Alabama, Tennessee, and Missouri.

IOWA DISTRIBUTION: map 11-b. SPECIMENS EXAMINED: Cass County, along U. S. Highway No. 71, near New Lyman, June 24, 1946, *Gilly 7346* (ISC). Johnson County, Iowa City, 188-, *Hitchcock* (ISC), June 7, 1895, *Shimek* (SUI), June 8, 1928, *Shimek* (SUI). Lee County, Keokuk, June 1, 1897, *Shimek* (ISC, SUI). Montgomery County, along U. S. Highway No. 71, just south of Villisca, June 25, 1946, *Gilly 7352* (ISC).

68. *Carex crus-corvi* Shuttlw. ex Kunze, Suppl. Riedgr. 128. 1842.

Raven's-foot sedge—swamps and river floodplains—Georgia and Florida to Texas, northward in the Mississippi Valley and Great Lakes Basin to Indiana, southern Michigan, Minnesota, and eastern Nebraska. IOWA DISTRIBUTION: map 11-e. UNCONFIRMED COUNTY REPORTS: Boone (141) and Muscatine (99, 107).

#### KEY TO IOWA CAREX—GROUP G

- A. Perigynia either very obscurely nerved, or conspicuously 2-keeled but otherwise nerveless or very nearly so.
  - B. Spikes widely spreading or drooping on slender flexuous peduncles; perigynia glabrous, shining; beaks conical-subulate, as long as or longer than the perigynium bodies .....69. *C. sprengelii*
  - BB. Spikes sessile or erect on firm peduncles; perigynia puberulent or pubescent, dull-surfaced; beaks shorter than perigynium-bodies.
    - C. Perigynia conspicuously 2-keeled, otherwise nerveless, puberulent or short-pubescent.
      - D. Fertile culms (those bearing spikes) all alike, more or less erect and elongated and bearing both staminate and pistillate flowers.
        - E. Bodies of the perigynia (both beak and stipitate base excluded) oblong-ovoid in shape, considerably longer than the diameter .....70. *C. artitecta*
        - EE. Bodies of the perigynia (both beak and stipitate base excluded) essentially globose, scarcely (if at all) longer than the diameter.
          - F. Plants caespitose, but not spreading by long-creeping rhizomes; lowest inflorescence bract either green and leaf-like or bifid or truncate and the midrib prolonged as an awn .....71. *C. communis*
          - FF. Plants sub-caespitose, spreading by long-creeping rhizomes; lowest inflorescence bract more or less scale-like, or longer and gradually tapering to the apex, rarely green and leaf-like and not awned.
            - G. Perigynium body, excluding beak and stipitate base, globular and about 1.5 mm. in diameter; usually found in woodlands .....72. *C. pensylvanica*
            - GG. Perigynium body, excluding beak and stipitate base, broadly ellipsoid, 2.25 mm.–2.75 mm. long and 2 mm.–2.25 mm. in diameter; usually found on dry prairies.....72a. *C. pensylvanica* var. *digyna*
    - DD. Fertile culms of two sorts, some elongated and bearing staminate spikes (or very rarely also an occasional pistillate spike), the others short, more or less hidden in the leaves, and bearing only pistillate spikes .....73. *C. tonsa*
  - CC. Perigynia not 2-keeled, nerveless or obscurely nerved, very conspicuously pubescent.
    - D. Foliage and culms pubescent; pistillate spikes loosely few-flowered; scales ciliate-margined .....forms of 43. *C. hirtifolia* (in Group D)
    - DD. Foliage and culms glabrous; pistillate spikes closely many-flowered; scales entire or hyaline and erose but not ciliate-margined.
      - E. Culms usually smooth; leaves 2 mm. or less in width, margins involute toward apex; perigynia oblong-ovoid or ellipsoid, 3 mm.–5 mm. long, 1.75 mm. thick; style bent or twisted above achene...74. *C. lasiocarpa* var. *americana*

- EE. Culms usually rough; leaves 1.5 mm.-5 mm. wide, margins revolute toward apex; perigynia broadly ovoid or obovoid, 2.5 mm.-3.5 mm. long, 1.75 mm.-2 mm. thick; style straight above  
achene ..... 74a. *C. lasiocarpa*  
var. *latifolia*
- AA. Perigynia conspicuously numerous-nerved (the nerves spaced more or less uniformly around the diameter of the perigynia), not 2-keeled.
- B. Spikes widely spreading or drooping on slender flexuous peduncles.
- C. Beaks of perigynia with rigid erect teeth 0.5 mm.  
in length ..... 75. *C. hystericina*
- CC. Beaks of perigynia with recurved-spreading teeth 1.25 mm.-2 mm.  
in length ..... 76. *C. comosa*
- BB. Spikes either sessile or stiffly erect (or somewhat spreading) on firm short peduncles.
- C. Perigynia sub-coriaceous and firm-walled, rather closely surrounding the achenes and scarcely (if at all) inflated.
- D. Perigynia pubescent ..... 77. *C. trichocarpa*
- DD. Perigynia glabrous.
- E. Teeth of perigynium-beaks 0.5 mm. in length, blunt and stiffly erect ..... 78. *C. lacustris*
- EE. Teeth of perigynium-beaks 1 mm.-3 mm. long, acute-tapering and spreading or recurved.
- F. Leaf blades and leaf sheaths pubescent, the sheaths brownish or purplish at mouth; perigynia lanceolate or ovoid-lanceolate, the teeth of the bifurcate beak usually 2 mm.-3 mm. long ..... 79. *C. atherodes*
- FF. Leaf blades and leaf sheaths glabrous, the sheaths hyaline or greenish at mouth; perigynia broadly ovoid, the teeth of the bifurcate beak usually 1 mm.-2 mm. long ..... 79a. *C. atherodes*  
var. *longo-lanceolata*
- CC. Perigynia membranous and thin-walled, considerably inflated, each achene thus filling only a portion of the perigynium which surrounds it.
- D. Perigynia less than 10 mm. in length.
- E. Scales of the pistillate flowers acute, acuminate, or mucronate, the midribs (except rarely in a few of the lowermost flowers in each spike) not prolonged into scabrid awns.
- F. Bracts subtending the pistillate spikes shorter than or only somewhat exceeding the entire inflorescence; perigynia ascending or somewhat spreading.
- G. Perigynia ellipsoid, broadest about the middle of the bodies, more or less tapering toward the base, 2 mm.-3.5 mm. thick.
- H. Base of the culms scarcely (if at all) spongy-inflated; pistillate spikes somewhat loosely flowered with 30-100 perigynia arranged in 5-8 rows ..... 80. *C. vesicaria*
- HH. Base of culms considerably spongy-inflated; pistillate spikes densely and closely-flowered with 40-450 perigynia crowded into many rows ..... 81. *C. rostrata*  
var. *utriculata*
- GG. Perigynia ovoid, broadest at the base, 4 mm.-6.5 mm. thick ..... 82. *C. tuckermanni*
- FF. Bracts subtending the pistillate spikes with well-developed blades, several to many times exceeding the whole inflorescence in length; perigynia wide-spreading, many of the lower ones reflexed ..... 83. *C. retrorsa*
- EE. Scales of the pistillate flowers with midribs prolonged as long scabrid awns ..... 84. *C. lurida*
- DD. Perigynia 10 mm. or more in length.
- E. Pistillate spikes globose or sub-globose; style straight for its entire length ..... 85. *C. grayii*
- EE. Pistillate spikes oblong-cylindrical or cylindrical; style bent or twisted just above achene.
- F. Achenes longer than thick, the sides shallowly concave, the three angles knobless ..... 86. *C. lupulina*
- FF. Achenes as thick or thicker than their length, the sides deeply concave, the three angles prominently knobbed ..... 87. *C. lupuliformis*



69. *Carex sprengelii* Dewey ex Spreng. Syst. 3:827. 1826.

*C. longirostris* Torr. ex Schw. Ann. Lyc. N. Y. 1:71. 1824; not of Krock. 1814 (B, BB, G).

Sprengel's sedge; long-beaked sedge—wooded slopes and rocky ledges, occasionally along ponds and streams—New Brunswick to Ontario and Alberta, south to Delaware, Pennsylvania, Indiana, Iowa, and Colorado.

IOWA DISTRIBUTION: map 11-f. UNCONFIRMED COUNTY REPORTS: Clinton (107), Dickinson (157), Floyd (101), and Scott (99, 107).

70. *Carex artitecta* Mackenz. N. Amer. Fl. 18:189. 1935.

*C. varia* Muhl. ex Wahl. Sv. Vet.-Akad. Nya. Handl. 24:159. 1803; not of Lumnitzer 1791; nor of Host. 1801.

Protected sedge—usually in dry soil, frequently along lake shores, sometimes in sandy soil—Vermont to Ontario, Michigan, and Iowa, south to South Carolina, Tennessee, Arkansas, and Oklahoma.

IOWA DISTRIBUTION: map 11-g. SPECIMENS EXAMINED: Emmet County, High Lake, June 11, 1917, Wolden 302 (ISC, W); Mud Lake, May 6, 1922, Wolden 381 (ISC, W). Fremont County, Tabor, 1898, Baldwin (ISC).

71. *Carex communis* L. H. Bailey, Mem. Torrey Club 1:41. 1889.

*C. varia* var. *pedicellata* Dewey, Amer. Jour. Sci. 11:163. 1826.

*C. pedicellata* (Dewey) Britt. ex L. H. Bailey, Mem. Torrey Club 5:87. 1894.

Fibrous-rooted sedge—dry woodlands, rocky hillsides, bluffs, and rocky ledges—Nova Scotia and Quebec to Minnesota, south to Georgia, Kentucky, and Arkansas.

IOWA DISTRIBUTION: map 11-h. SPECIMENS EXAMINED: Fayette County, Fayette, May 5, 1894, Fink (ISC). Madison County, Peru, June 20, 1897, Hollingsworth (G, ISC). Muscatine County, Muscatine, without date, Ball (ISC); Wild Cat Den State Park, June, 1897, Barnes, Miller, and Martin (ISC, NY, SUI). UNCONFIRMED COUNTY REPORT: Scott (107).

72. *Carex pensylvanica* Lam. Encyc. 3:388. 1791.

Pennsylvania sedge—open woodlands and woodland edge thickets—Nova Scotia and Quebec to Minnesota and North Dakota, south to South Carolina, Tennessee, Missouri, and Iowa.

IOWA DISTRIBUTION: map 11-i. UNCONFIRMED COUNTY REPORTS: Allamakee (160), Floyd (107), Kossuth (107), Lee (121), Louisa (102), Monona (101), Scott (107), and Shelby (119). NOTES: (a) At least some of these reports must refer to the var. *digyna*, rather than to the typical phase of the species. (b) As originally published, the specific epithet was spelled with only one *n*; therefore, the spelling as given here should be used. Fernald (31a) and Egler (21a) have adequately discussed the problems posed by the variation in spelling of this epithet.

- 72a. *Carex pensylvanica* Lam., var. *digyna* Böckl. Linnaea 41:220. 1877.  
*C. heliophila* Mackenz. Torreyia 13:15. 1913.

Prairie Pennsylvania sedge; sunlight sedge—dry prairies and plains, often in gravelly or sandy soil—Manitoba to Alberta, south to Illinois, Missouri, Kansas, New Mexico, and Colorado.

IOWA DISTRIBUTION: map 11-j.

73. *Carex tonsa* (Fern.) Bickn. Bul. Torrey Club 35:492. 1908.  
*C. umbellata* var. *tonsa* Fern. Proc. Amer. Acad. 37:507. 1902.

Deep-green sedge—sandy soil and in sand dunes—Nova Scotia and Quebec to Minnesota and Alberta, south to Delaware, Maryland, Indiana, and Iowa.

IOWA DISTRIBUTION: map 11-g. SPECIMENS EXAMINED: Allamakee County, 5–10 miles west of New Albin, September 13, 1937, *Tolstead* (ISC); 4 miles south of New Albin, June 18, 1940, *Hayden* 9960 (ISC, NY).

74. *Carex lasiocarpa* Ehrh., var. *americana* Fern. Rhodora 44:304. 1942.  
*"C. filiformis"* of many authors; not of L. 1753 (B).  
*"C. lasiocarpa"* of most American authors; not of Ehrh. 1784.

Slender sedge—swamps, sphagnum bogs, sloughs, borders of lakes and ponds, prairie swales—Newfoundland to Keewatin and British Columbia, south to northern New Jersey, Pennsylvania, Ohio, Illinois, Iowa, Manitoba, Saskatchewan, and Washington.

IOWA DISTRIBUTION: map 11-k. UNCONFIRMED COUNTY REPORTS: Cerro Gordo (142), Shelby (119), Winnebago (142), and Worth (142). NOTE: Although usually recognized as a distinct species, *C. lanuginosa*, a more robust plant, seems to be merely the North American segment of a more robust phase of the species found toward the southern part of the species range in North America, Europe, and Asia; Clausen and Wahl (17a) have recognized the robust American plant as a subspecies. Fernald (32) has recently indicated that the typical form of this species is confined to northern Europe and Asia; for this reason he has separated the more slender American plants as var. *americana*. After studying a large series of specimens from the three continents I am more or less in agreement with Clausen and Wahl, although varietal status for both of the American plants seems preferable to recognizing them as subspecies. Further study of related species may result eventually in a reunion of the "*lasiocarpa-lanuginosa*" complex with the northern Eurasian *C. filiformis* L.

- 74a. *Carex lasiocarpa* Ehrh., var. *latifolia* (Böckl.) Gilly, comb. nov.<sup>21</sup>  
*C. lanuginosa* Michx. Fl. Bor.-Amer. 2:175. 1803.  
*C. filiformis* var. *latifolia* Böckl. Linnaea 41:309. 1877.

<sup>21</sup> Because of the necessity of using the oldest available varietal epithet—Article 58 of the *International Rules of Botanical Nomenclature* (12), which states: "... when a subdivision of a species becomes a species, or when the reverse of these changes take place, and in general when a group changes its rank, the earliest legitimate name or epithet given to the group in its new rank is valid, unless that name or the resulting association or combination is a later homonym . . ."—this combination must replace the already available *C. lasiocarpa* var. *lanuginosa* (Michx.) Kük.

*C. filiformis* var. *lanuginosa* (Michx.) B.S.P. Prel. Cat. N. Y. 63. 1888.  
*C. lasiocarpa* var. *lanuginosa* (Michx.) Kük. Pflanze. 38[IV. 20]:  
 748. 1909.

*C. lasiocarpa* ssp. *lanuginosa* (Michx.) Clausen & Wahl, Rhodora  
 41: 31. 1939.

Woolly sedge—habitat essentially the same as for the var. *americana*—New Brunswick, Quebec, and Ontario to Minnesota, Manitoba, Saskatchewan, Alberta, and British Columbia, south to Delaware, Virginia, Tennessee, Arkansas, Texas, New Mexico, Arizona, and California.  
 IOWA DISTRIBUTION: map 11-l. UNCONFIRMED COUNTY REPORTS: Henry (103), Linn (133), and Scott (99, 107).

75. *Carex hystericina* Muhl. ex. Willd. Sp. Pl. 4: 282. 1805.

"*C. hystericina*" of many authors (B, BB, BB2, R).

Porcupine sedge—swamps, wet prairies, swales, marshes, hanging bogs, and along stream banks and pond and lake shores, sometimes in shallow water—New Brunswick and Quebec to Manitoba, Alberta, and Washington, south to Virginia, Missouri, Oklahoma, Texas, New Mexico, and California.

IOWA DISTRIBUTION: map 12-a. UNCONFIRMED COUNTY REPORTS: Decatur (119), Fayette (126), Hardin (148), Lee (121), Linn (133), Mahaska (96), Muscatine (99, 107), Scott (99, 107), and Shelby (119). NOTE: Although the specific epithet is frequently given as "*hystericina*," the original spelling of the epithet when published is as given above; this original spelling must be retained.

76. *Carex comosa* Boott, Trans. Linn. Soc. 20: 117. 1846.

*C. pseudo-cyperus* var. *americana* Hochst. ex L. H. Bailey, Mem. Torrey Club 1: 54. 1889.

Bristly sedge—marshes, swamps, prairie sloughs, and in shallow water along lake shores—Maine and Quebec to Minnesota and Nebraska, south to Florida and Louisiana; also along the Pacific Coast from Washington to central California and inland to Idaho.

IOWA DISTRIBUTION: map 12-b. UNCONFIRMED COUNTY REPORTS: Hardin (148), Henry (103), Johnson (164), and Muscatine (107).

77. *Carex trichocarpa* Muhl. ex. Schkuhr, Riedgr. Nachtr. 47. 1806.

Hairy-fruited sedge—marshes, swales, swamps, and wet meadows—Vermont and Quebec to Minnesota, south to Delaware, Pennsylvania, Indiana, and Iowa.

IOWA DISTRIBUTION: map 12-c. UNCONFIRMED COUNTY REPORTS: Benton (107), Dickinson (157), Hardin (148), Lyon (153), Scott (99), and Story (107, 132).

78. *Carex lacustris* Willd. Sp. Pl. 4: 306. 1805.

"*C. riparia*" of numerous authors; not of Curtis. 1783 (B, BB, G).

River-bank sedge—swamps, hanging bogs, marshes, and along river and lake shores, sometimes in water up to 1 foot deep—Nova Scotia and Quebec to Manitoba, south to Maryland, Ohio, and Iowa.

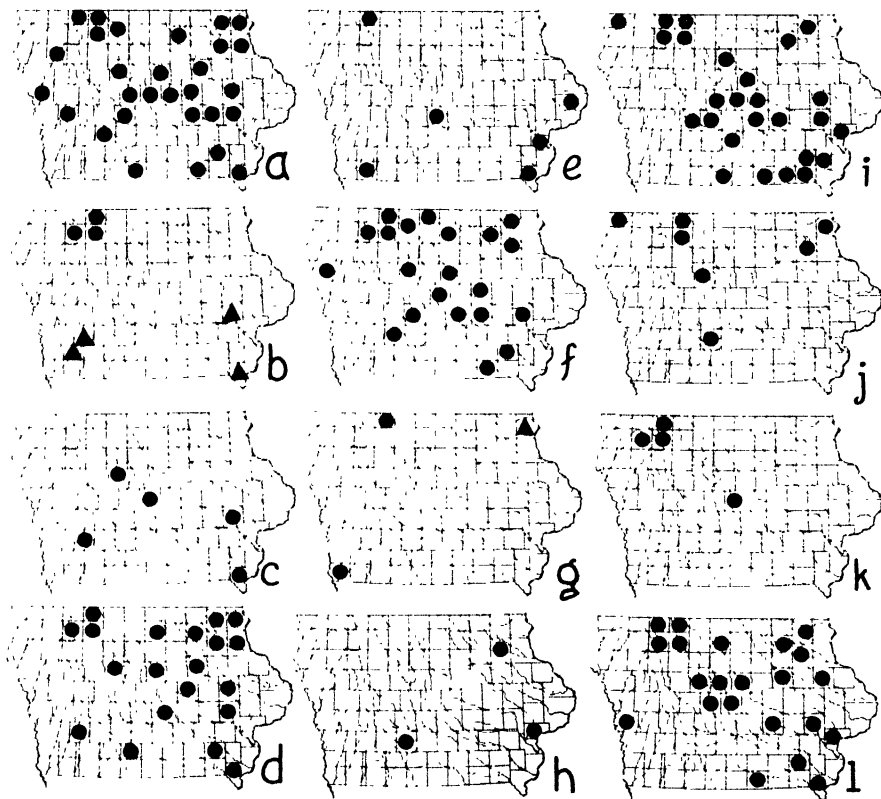


FIGURE 11. Distribution Maps of Iowa Cyperaceae.

- a—*Carex rosea* var. *pusilla*.
- b—*Carex alopecoidea* (•) and *Carex stipata* var. *laevivaginata* (▲).
- c—*Carex conjuncta*.
- d—*Carex stipata*.
- e—*Carex crus-corvi*.
- f—*Carex sprenkelii*.
- g—*Carex artitecta* (•) and *Carex tonsa* (▲).
- h—*Carex communis*.
- i—*Carex pennsylvanica*.
- j—*Carex pennsylvanica* var. *digyna*.
- k—*Carex lasiocarpa* var. *americana*.
- l—*Carex lasiocarpa* var. *latifolia*.

IOWA DISTRIBUTION: map 12-d. UNCONFIRMED COUNTY REPORT: Dickinson (143). NOTE: The report of this species from Black Hawk County (104) is based on a misidentification of a specimen of *C. atherodes*.

79. *Carex atherodes* Spreng. Syst. 3:827. 1826.

*C. aristata* R. Br. in Richards. in Frankl. Journey 751. 1823; not of Honck. 1792 (B).

*C. trichocarpa* var. *imberbis* A. Gray, Man. ed. 5. 597. 1867.

*C. trichocarpa* var. *aristata* (R. Br.) L. H. Bailey, Bot. Gaz. 10: 294. 1885.

Awned sedge—swamps, bogs, marshes, river banks, frequently in standing water up to at least 18 inches deep—Ontario to Mackenzie, Yukon, and British Columbia, south to western New York, Indiana, Missouri, Kansas, Colorado, and Oregon.

IOWA DISTRIBUTION: map 12-e. UNCONFIRMED COUNTY REPORTS: Dallas (107), Hardin (148), Henry (103), and Mahaska (96). NOTE: Intermediates, on the basis of several combinations of characters, between this species and the following variety are of frequent occurrence; in this treatment the individual intermediate specimen has been assigned to either the species or the variety on the basis of the proportion of the two sets of characteristics which it possessed.

79a. *Carex atherodes* Spreng., var. *longo-lanceolata* (Dewey) Gilly, comb. nov.<sup>22</sup>

*C. aristata* var. *longo-lanceolata* Dewey, Amer. Jour. Sci. II 18:102. 1854.

*C. laeviconica* Dewey, Amer. Jour. Sci. II 24:47. 1857.

*C. trichocarpa* var. *deweyi* L. H. Bailey, Bot. Gaz. 10:293. 1885.

*C. trichocarpa* var. *laeviconica* (Dewey) Hitchc. Trans. Acad. St. Louis 5:524. 1891.

*C. aristata* ssp. *trichocarpa* var. *laeviconica* (Dewey) Kük. Pflanzenr. 38[IV. 20]:754. 1939.

Smooth-sheathed awned sedge—habitat essentially the same as for the species—Manitoba to Saskatchewan, south to Illinois, Missouri, Kansas, South Dakota, and Montana.

IOWA DISTRIBUTION: map 12-f. UNCONFIRMED COUNTY REPORT: Muscatine (107).

80. *Carex vesicaria* L. Sp. Pl. 979, in part. 1753.

*C. monile* Tuckerm. Enum. Caric. 20. 1843 (B, BB).

*C. vesicaria* var. *monile* (Tuckerm.) Fern. Rhodora 3:53. 1901.

Inflated sedge—swampy or marshy places, low wet prairies, and pond or lake edges—Newfoundland to Keewatin and British Columbia, south to Delaware, Indiana, Missouri, N. Mexico, and California; also widely distributed in Eurasia and reported from north Africa.

<sup>22</sup> Although the varietal epithets *deweyi* and *laeviconica* would seem to be available for this variety, because of Article 58 of the *International Rules of Botanical Nomenclature* (12), this combination is necessary. See also footnote 21 of this paper.

IOWA DISTRIBUTION: map 12-g. UNCONFIRMED COUNTY REPORTS: Hardin (148), Muscatine (99, 107), Poweshiek (94), and Scott (99, 107).

81. *Carex rostrata* Stokes, var. *utriculata* (Boott) L. H. Bailey, Proc. Amer. Acad. 22:67. 1886.

"*C. inflata*" of many authors; not of Huds. 1762 (*mixtum*), nor 1778. "*C. rostrata*" of many American authors; not of Stokes 1787 (BB2, G, R).

*C. utriculata* Boott in Hook. Fl. Bor.-Amer. 2:221. 1839 (B, BB).

*C. inflata* var. *utriculata* (Boott) Druce, Bot. Soc. & Exchange Club Brit. Isl. Rept. 9:141. 1930.

Bottle sedge—swamps, marshes, bogs, pond and lake edges, sometimes in water up to at least one foot deep—Labrador and Newfoundland to British Columbia, south to Delaware, District of Columbia, West Virginia, Ohio, Indiana, Iowa, South Dakota, New Mexico, and California. IOWA DISTRIBUTION: map 12-h. NOTES: (a) Fernald (32) discussed the status of this species four years ago. At that time he found it necessary to accept the apparent priority of *C. inflata* and at the same time he recognized that much of the American material of the species belonged to this variety rather than the typical phase of the species which is common in Eurasia and ranges across North America somewhat to the north of the distributional range of this variety. (b) After this paper had been accepted for publication but, fortunately, before the typescript had gone to the printer, Fernald's revised opinion (34a) of the correct name for this species appeared in print. Accordingly, the change from *C. inflata* back to *C. rostrata* has also been made here. It seems relatively safe to follow Fernald in this nomenclatural regression inasmuch as his decision is primarily based on evidence presented by Nelmès (59a).

82. *Carex tuckermanni* Boott ex Dewey, Amer. Jour. Sci. 49:48. 1845.

Tuckerman's sedge—swampy woodlands and meadows—New Brunswick to Ontario and Minnesota, south to New Jersey, Ohio, and Iowa.

IOWA DISTRIBUTION: map 12-h. SPECIMENS EXAMINED: Jasper County, Richland Township, 1886, Norris (GRC), August 20, 1897, Norris (ISC).

UNCONFIRMED COUNTY REPORT: Poweshiek (107).

83. *Carex retrorsa* Schw. Ann. Lyc. N. Y. 1:71. 1824.

Retorse sedge—swampy woodlands, marshes, and floodplains—Nova Scotia and Quebec to Saskatchewan and British Columbia, south to New Jersey, Ohio, Iowa, South Dakota, Colorado, and Oregon.

IOWA DISTRIBUTION: map 12-i. UNCONFIRMED COUNTY REPORT: Johnson (164). NOTE: A report of this species from Lee County (121) was based on a misidentification of a specimen of *C. lurida*.

84. *Carex lurida* Wahl. Sv. Vet.-Akad. Nya Handl. 24:153. 1803.

*C. lurida* var. *parvula* L. H. Bailey, Bul. Torrey Club. 20:418. 1893.

Sallow sedge—swamps, wet meadows, prairies, and ravines—Nova Scotia and Quebec to Minnesota and Oklahoma, south to Florida, Louisiana, and Texas; south along the Gulf Coast into eastern Mexico.

IOWA DISTRIBUTION: map 12-j. SPECIMENS EXAMINED: Jefferson County, Lockridge Township, August 12, 1935, McDonald 2752 (P). Lee County, July 9, 1931, Fults 1330 (ISC). Story County, Ames, 1886, Hitchcock (ISC). UNCONFIRMED COUNTY REPORT: Dickinson (89).

85. *Carex grayii* Carey, Amer. Jour. Sci. II 4:22. 1847.

"*C. folliculata*" of Iowa authors; not of L. 1753.

*C. grayii* var. *hispidula* A. Gray ex L. H. Bailey, Mem. Torrey Club 1: 54. 1889.

*C. asa-grayii* L. H. Bailey, Bul. Torrey Club 20:427. 1893 (B, BB, BB2, R).

*C. asa-grayii* var. *hispidula* (A. Gray) L. H. Bailey, l. c.

Gray's sedge—low wet woodlands and floodplain marshes—Newfoundland to Keewatin and Minnesota, south to Florida, Louisiana, and Texas.

IOWA DISTRIBUTION: map 12-k. UNCONFIRMED COUNTY REPORTS: Cerro Gordo (142), Hamilton (142), Henry (103), Mahaska (96), Muscatine (99, 107), Scott (99, 107), Shelby (107), Winnebago (142), Winneshiek (124), Worth (142), and Wright (142). NOTE: The doubtfully distinct hispid-fruited phase (the var. *hispidula*) has been collected in Jasper County and may be more widely distributed in the southeastern part of the state.

86. *Carex lupulina* Muhl. ex Willd. Sp. Pl. 4:266. 1805.

*C. lupulina* var. *pedunculata* A. Gray ex Beck, Bot. U. S. 438. 1833.

Hop sedge—marshes, swamps, low swampy woodlands, and stream floodplains—Nova Scotia to Ontario and Minnesota, south to Florida, Texas, and Oklahoma.

IOWA DISTRIBUTION: map 12-l. UNCONFIRMED COUNTY REPORTS: Adams (120), Clinton (107), Hamilton (142), Hardin (148), Henry (103), Mahaska (96), Montgomery (120), Scott (99, 107), and Union (120). NOTE: A report of this species from Decatur County (87) is based on a misidentification of a specimen of *C. lupuliformis*.

87. *Carex lupuliformis* Sartw. ex Dewey, Amer. Jour. Sci. II 9:29. 1850.

Hop-like sedge—swampy woodlands and low wet ground—Vermont to Ontario, Wisconsin, and Iowa, south to Delaware, Kentucky, Louisiana, Texas, and Oklahoma.

IOWA DISTRIBUTION: map 12-i. SPECIMENS EXAMINED: Decatur County, July 8, 1900, Anderson (ISC). Lee County, July 29, 1931, Fults 1644 (ISC); Keokuk, June 1, 1897, Shimek (SUI). UNCONFIRMED COUNTY REPORT: Mahaska (97).

#### SPECIES OF CAREX ERRONEOUSLY OR DOUBTFULLY REPORTED FOR IOWA

*Carex adusta* Boott—This species was reported from Iowa, without definite locality (90), and from Lyon County (151) as "*adusta* (?)." Cratty (107), under *C. foenea perplexa* on page 360, states: "Spirit Lake, June 24, 1881, Arthur. This latter is the *C. adusta* of Arthur's Catalogue."

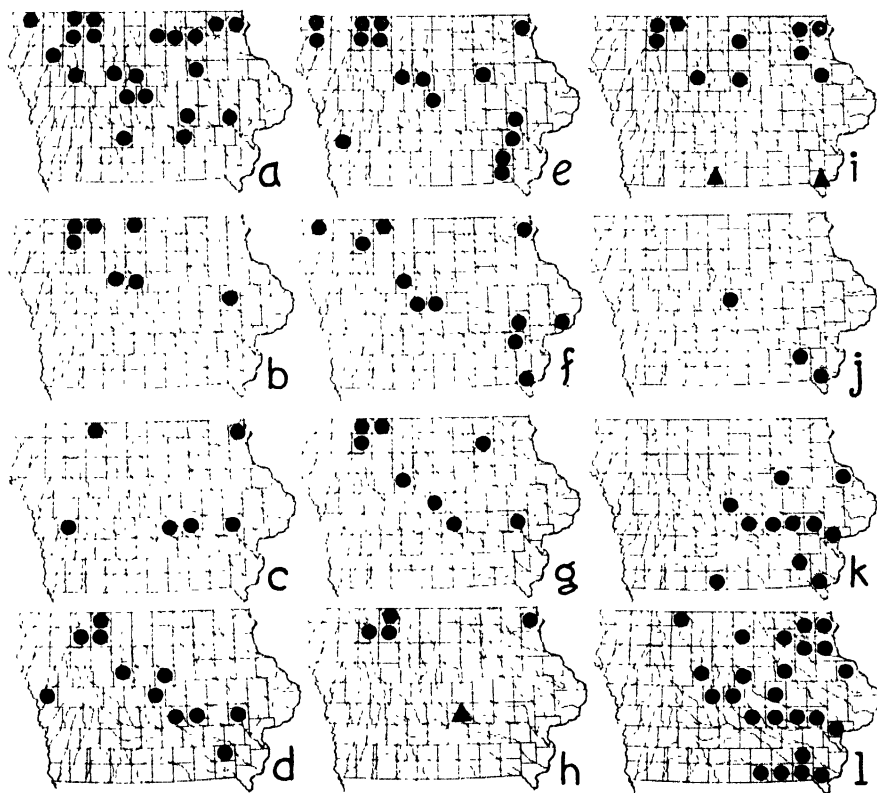


FIGURE 12. Distribution Maps of Iowa Cyperaceae.

- a—*Carex hystericina*.
- b—*Carex comosa*.
- c—*Carex trichocarpa*.
- d—*Carex lacustris*.
- e—*Carex atherodes*.
- f—*Carex atherodes* var. *longo-lanceolata*.
- g—*Carex vesicaria*.
- h—*Carex rostrata* var. *utriculata* (•) and *Carex tuckermanni* (▲).
- i—*Carex retrorsa* (•) and *Carex lupuliformis* (▲).
- j—*Carex lurida*.
- k—*Carex grayii*.
- l—*Carex lupulina*.



and on page 369: "*C. adusta* Boott. Prof. Shimek collected at Rock Rapids, Lyon Co., Aug. 1896, what appears to be this species. Further observation is needed." Both the Arthur and Shimek specimens mentioned by Cratty are representative of *C. brevior* (Dewey) Mackenz. as accepted in this paper. See also discussion of *C. foenea* var. *perplexa* below.

*Carex aenea* Fern.—The report of this species from Mahaska County (96) has not been confirmed, but it is almost certainly based on a misidentification of some member of the section *Ovales* (see page 96 of this paper). *C. aenea*, a northern species, is known from Minnesota, Wisconsin, and South Dakota, as well as further eastward.

*Carex alata* Torr.—The reports of this species from Iowa, without exact locality (90, 107) and from Ames, Story County—as *C. straminea* var. *alata* (Torr.) L. H. Bailey—(107, 132) are based on specimens of *C. bicknellii* Britt. The reports from Poweshiek County (105, 107) are based on a specimen of *C. suberecta* (Olney) Britt.

*Carex aperta* Boott.—The report of this species for Iowa, without exact locality (90), apparently refers to *C. haydenii* Dewey.

*Carex arcta* Boott.—I have not seen the specimen on which the report of this species from Mahaska County (96) is based. At present the known range of this species is from New Brunswick and Quebec to Alberta and British Columbia, south to Massachusetts, New York, Michigan, Minnesota, Idaho, and California, and it seems doubtful that it occurs in Iowa.

*Carex bromoides* Schkuhr.—The report of this species for Story County (100) has not yet been substantiated.

*Carex castanea* Willd.—The report of this species from the Grinnell area (105) has not yet been confirmed.

*Carex digitalis* Willd.—The report of this species from Johnson County (164) is apparently based on a specimen of *C. laxiculmis* Schw.

"*Carex foenea*" of authors (= *C. argyrantha* Tuckerm.); not *C. foenea* Willd.—The reports of this species from Scott County (99, 107) and Story County (107) are based on a series of specimens of *C. bicknellii* Britt., and the report of the species for Iowa in the *Illustrated Flora* (16) has not yet been confirmed. See page 98 of this paper for a discussion of the true *C. foenea*.

*Carex foenea* var. *perplexa* L. H. Bailey.—The report of this variety from Scott County (107) is based on a mixture of *C. festuacea* Schkuhr and *C. brevior* (Dewey) Mackenz., and the reports from Dickinson County (107, 157) are based on specimens of *C. brevior*. See also the discussion under *C. adusta* above.

*Carex formosa* Dewey.—The report of this species from Black Hawk County (104) was based on a specimen of *C. davisii* Schw. & Torr. and the report from Decatur County (110) is based on a specimen of *C. shortiana* Dewey.

*Carex hormathodes* Fern.—This species is listed for Iowa in "Gray's"

*Manual* (66) and in the *Illustrated Flora* (16). The report is still unconfirmed and is probably based on a misidentification.

*Carex houghtonii* Torr.—The report of this species from "Council Bluffs, Iowa" by Upham (82) has not yet been substantiated.

*Carex hyalinolepis* Steud.—The report of this species for Iowa, without exact locality (53) is based upon specimens which I am obliged to place in *C. lacustris* Willd.

*Carex intumescens* Rudge.—The reports of this species, without exact locality (53), from central Iowa (106) and from Dubuque County (118) are based on specimens of *C. grayii* Carey. It might be noted here that an eventual reevaluation of the relationship between *C. grayii* and *C. intumescens* may result in the merging of these two species.

*Carex leptalea* Wahl.—This species is known from the neighboring states of Minnesota, Wisconsin, Illinois, and Missouri. It may, therefore, be expected in eastern Iowa. For this reason the species has been inserted in the key for the genus *Carex* in this paper (see page 105).

*Carex lupulina* var. *bella-villa* L. H. Bailey (= *C. macounii* Dewey) —The report of this variety from Hardin County (148) is based on an aberrant specimen of *C. retrorsa* Schw. Mackenzie (53) considered this variety as one of the several phases of a complex series of hybrids in which *C. lupulina*, *C. retrorsa* and a number of other species were involved.

*Carex michauxiana* Böckl.—The report of this species from the Grinnell area (105) has not yet been substantiated.

*Carex pauciflora* Lightf.—The report of this species for Emmet County (142) has not been substantiated. Perhaps it is merely a typographical error for *Eleocharis pauciflora* (Lightf.) Link.

*Carex plantaginea* Lam.—The report of this species for Lee County (90) has not yet been confirmed. It may, perhaps, be based on a specimen of *C. albursina* Sheldon which superficially resembles *C. plantaginea*.

*Carex pseudo-cyperus* L.—The reports of this species from Dickinson and Emmet Counties (95) are based on specimens of *C. hystericina* Muhl.; the report from Hardin County (148) is based on a specimen of *C. comosa* Boott.

*Carex scoparia* var. *minor* Boott (= *C. crawfordii* Fern.)—The report of this variety from Iowa, without exact locality (90), has not been substantiated.

*Carex stellulata* Gooden (= *C. muricata* L.)—The reports of this species for Iowa, without exact locality (94), and from the Grinnell area (105) have not been substantiated.

*Carex sterilis* Willd.—The report of this species from Dallas County (107) is based on a misidentification of *C. muhlenbergii* Schkuhr; the reports from Lee County (107) and Scott County (99, 107) are based on misidentifications of *C. leavenworthii* Dewey.

*Carex straminea* Willd.—The report of this species from Decatur County (107) is based on a specimen of *C. normalis* Mackenz.; the report from Emmet County (107) is based on a specimen of *C. suberecta* (Olney)

Britt.; the reports from Lyon County (107, 151) are based on a specimen of *C. brevior* (Dewey) Mackenz.; and the reports from Scott County (99, 107) are based on specimens of *C. festucacea* Schkuhr. The reports from Boone County (141) and Linn County (133) have not been substantiated.

*Carex straminea* var. *alata* (Torr.) L. H. Bailey—see discussion under *C. alata*, above.

*Carex straminea* var. *aperta* Boott (= *C. richii* (Fern.) Mackenz.)—The reports of this variety from Johnson County (117) and Winneshiek County (117) have not been confirmed. Almost certainly they refer to some other species of the section *Ovales*. See discussion under *C. aenea*, above.

*Carex straminea* var. *hyalina* (Boott) Boott (= *C. hyalina* Boott)—The report of this variety from Iowa, without exact locality (90), is based on a specimen of *C. bicknellii* Britt. in the Gray Herbarium (G): June 3, 1880, Jones.<sup>23</sup>

*Carex tenella* Schkuhr (= *C. disperma* Dewey)—The author has not seen the specimen, on which the report of this species from Lee County (121) is based. Although this northern-ranging species is known from both Minnesota and Wisconsin, it is doubtful if it occurs in Iowa, particularly in the southeastern corner of the state.

*Carex torta* Boott—The report of this species from the Grinnell area (105) has not yet been substantiated.

*Carex triceps* var. *smithii* L. H. Bailey (= *C. caroliniana* Schw.)—The report of this species for Van Buren County (108) is based upon a specimen of *C. hirsutella* Mackenz.

*Carex trisperma* Dewey—The report of this species from Emmet County (142) is still unsubstantiated.

*Carex umbellata* Schkuhr—I have not seen the specimen upon which the report from Lee County (121) is based. The species is known from Illinois and Minnesota and might possibly be expected in eastern Iowa.

#### GENERA FREQUENTLY REPORTED FOR IOWA

*Fuirena* Rottb. Descr. et Ic. Pl. 70. 1773.—*F. squarrosa* Michx. has been reported from Johnson County (164), and one or more of the species of the genus are given a range in the regional manuals (14, 16, 66, 67) which includes Iowa. Thus far, I have been unable to find the specimen on which the Johnson County report was based; moreover, he has been unable to find any specimen of this genus which has been collected in Iowa. This genus is included in the Key to the Genera in this paper (see page 68) for the convenience of the student who might be so fortunate as to rediscover the genus within the state.

*Cladium* P. Br. Civ. & Nat. Hist. Jam. 114. 1756 [*Mariscus* (Hall)]

<sup>23</sup> The date on the specimen label is "Jan. 3, 1880," but that seems scarcely possible in view of the good condition of the specimen.

Zinn, Cat. Hort. Gott. 79. 1757]—*Cladium mariscoides* (Muhl.) Torr. [*Mariscus mariscoides* (Muhl.) Kuntze] is reported for Iowa in all the regional manuals. Thus far the author has been unable to locate a specimen of the species which has been collected in Iowa. Nevertheless, as with *Fuirena*, the genus is included in the Key to the Genera of this paper (see page 69).

## GLOSSARY

This list of terminology definitions should be used in conjunction with Figure 3 (page 67). It should be noted that no attempt has been made to give a complete and all-inclusive definition for any term included below. The aim has been, rather, to provide a brief and accurate explanation of each term in the sense in which it has been used in this particular paper (see also the discussion, page 57, of the necessity for using a specialized terminology in the family Cyperaceae).

- achene*—the cyperaceous fruit; a dry, indehiscent, hard-walled, 1-celled and 1-seeded structure.
- acuminate*—gradually narrowed and constricted to a sharp point (Fig. 3, N-5).
- acute*—abruptly and sharply pointed (Fig. 3, N-3).
- adnate*—attached by one side to another structure.
- androgynous spike* (or *spikelet*)—one with staminate flowers at apex and pistillate flowers below (see also gynecandrous spike).
- apiculation*—a terminal point or projection remaining on the achene after the deciduous style has fallen off (Fig. 3, H). This is of the same color and texture as the achene (compare with *tubercle*).
- appressed*—closely fitted together (but not united or fused) for total length.
- approximate*—attached close together (or at the same point) but not united or fused together.
- aristate*—awn-tipped (Fig. 3, N-6). This term should not be confused with *mucronate*.
- ascending*—erect, or pointed upwards.
- awn*—an elongated extension of the midrib beyond the margin of a leaf, bract or scale (Fig. 3, N-6).
- beak*—the constricted apical portion (or mouth) of a perigynium (Fig. 3, Q, W).
- biconvex*—having two opposite convexly-curved surfaces in cross-section (Fig. 3, M-1).
- bidentate*—two-toothed (Fig. 3, Q).
- bidentulate*—minutely two-toothed.
- bifid*—separated into two parts by a median slash- or wedge-shaped sinus (Fig. 3, N-7).
- bisexual flower*—a flower containing both stamens and pistil, i.e., a perfect flower (Fig. 3, F).
- bisexual spike*—a spike containing both staminate and pistillate flowers.
- body*—the expanded portion of a scale (as contrasted to a terminal awn) or of a perigynium (as contrasted to the narrowed or constricted beak).
- bracts*—reduced leaves which subtend individual spikes or clusters of spikes or spikelets in the sedge inflorescence. They may be leaf-like or scale-like in appearance.
- capillary*—thread-like (more delicate and slender than *filiform*).
- cauline leaves*—leaves attached at intervals to a stem-like culm, as contrasted to basally-clustered leaves.
- cespitose*—a plant with clustered or tufted stems; tussock-like.
- ciliate*—with a marginal fringe or row of hairs.
- cross-rugulose*—transversely wrinkled.
- culms*—the scape-like and essentially leafless aerial stems which support the inflorescence of the Cyperaceae.
- cuspidate*—with a broadly toothed apex; similar to *mucronate* except that the mucro is essentially wing-margined.
- dioecious*—a plant which bears *only* staminate flowers or *only* pistillate flowers, the other type of flowers being borne on another individual plant (see also *monoeious*).

- dorsal**—the outer ("back" or "lower") surface of an achene, perigynium, leaf blade, or leaf sheath; the surface which is furthest away from axis or culm when the structure under consideration is in an upright position (Fig. 3, O, P—*dor*).
- ellipsoid**—a solid object, such as an achene or a spikelet, with an elliptical profile.
- elliptical**—a flat-surface shape, broadest in the middle and gradually narrowing toward both ends (Fig. 3, L-5).
- erose**—with a ragged or very unevenly toothed margin or apex.
- filiform**—slender, frequently wiry, but not quite thread-like (somewhat coarser than capillary).
- flaccid**—limp, flabby; usually applied to leaves, sometimes to stems.
- glabrate**—becoming hairless.
- glabrous**—hairless.
- globose**—essentially spherical.
- gynecandrous spike (or spikelet)**—one with pistillate flowers at apex and staminate flowers below (see also androgynous).
- hispid**—covered with stiff hairs or bristles, thus apparently harsh and rough surfaced.
- hispidulous**—covered with minute stiff hairs.
- hyaline**—colorless or translucent, usually also thin.
- hypogynium**—a basal "cup" in which the achenes of *Scleria* are borne (Fig. 3, V).
- hypogynous**—a flower with all other flower parts (stamens, perianth, etc.) attached below the pistil (gynoecium).
- imbricate**—overlapping, more or less like shingles on a roof (Fig. 3, B).
- impressed**—sunken below the surface.
- inflorescence**—the total aggregation of spikes (or spikelets), their supporting pedicels and subtending bracts, of a single Cyperaceous plant.
- involute**—inrolled, as the margins of a leaf rolling or bending in toward the midrib of the upper surface of the leaf (see also revolute).
- lanceolate**—a narrow flat-surface shape, broadest below the middle and rather gradually narrowed to apex (Fig. 3, L-3); compare with *ovate* and *oblanceolate*.
- lax**—loose-spreading, not stiffly erect; when applied to inflorescences this term indicates that the individual spikes or spikelets are rather widely separated from each other.
- lenticular**—biconvex (Fig. 3, M-1).
- ligule**—the upward-projecting portion of the leaf-sheath lining visible at the mouth of the sheath and across the base of the leaf blade (Fig. 3, X—*lig*).
- moniliform inflorescence**—an inflorescence which resembles a string of beads, the spikes (or spikelets), therefore, somewhat separated and more or less equidistant.
- monoecious**—a plant bearing both staminate and pistillate flowers (see also dioecious).
- mucro**—a short, sharp, or blunt projection of the midrib of a leaf or scale beyond the margin (Fig. 3, N-4).
- mucronate**—tipped with a mucro; this term should not be confused with *aristate*.
- nerves**—the small vascular bundles visible in a leaf blade, leaf sheath, scale or perigynium (sometimes also called "veins" or "ribs").
- oblanceolate**—a narrow flat-surface shape, broadest above the middle and gradually narrowing toward the base (Fig. 3, L-6); compare with *obovate* and *lanceolate*.
- oblong**—an essentially rectangular flat-surface shape (Fig. 3, L-2).
- obovate**—a wide flat-surface shape, broadest above the middle (Fig. 3, L-7); compare with *oblanceolate* and *ovate*.
- obovoid**—a solid object, such as an achene or spikelet, having an obovoid profile.
- obtuse**—abruptly blunt-pointed or rounded at the apex (Fig. 3, N-2).
- orbicular**—almost circular; a flat-surface shape (Fig. 3, L-8).
- ovate**—a wide flat-surface shape, broadest below the middle (Fig. 3, L-4); compare with *lanceolate* and *obovate*.
- ovoid**—a solid object, like an achene or a spikelet, with an ovoid profile.
- papillose**—the surface covered with small rounded protuberances; minutely warty.
- pedicel**—the stalk on which an individual flower or spikelet is attached.
- peduncle**—the common stalk on which a number of flowers or spikelets are attached.
- perfect flower**—a flower containing both stamens and pistil (i.e., bisexual).
- perigynium**—a modified leaf sheath or prophyll which surrounds the pistillate flower, and the resultant achene, in the genus *Carex* (Fig. 3, D, Q, S, W).
- pistillate flower**—a unisexual flower containing only the pistil (gynoecium) and its appendages; small, abortive and nonfunctional stamens are sometimes present (see also staminate flower).
- plano-convex**—a cross-section shape in which a flat surface is opposite to a convexly-curved surface (Fig. 3, M-2).

- pubescent*—hairy; the surface covered with hairs.  
*punctulate*—the surface minutely dotted.  
*rachilla*—a secondary axis in a compound inflorescence; *rachillae*—the plural.  
*rachis*—the primary axis in a compound inflorescence.  
*recurved*—gradually or only partially bent downward.  
*reflexed*—sharply and abruptly bent downward, usually appressed against the supporting stalk.  
*retorse*—bent toward the base of the organ or structure to which attached.  
*revolute*—out-rolled, as the margins of a leaf rolling or bending in toward the midrib of the lower surface of the leaf (*see also* involute).  
*rhizomatous*—having rhizomes (modified and usually underground stems which are frequently mistaken for roots).  
*rugose*—with a wrinkled surface.  
*rugulose*—with the surface minutely wrinkled.  
*scabrid*; *scabrous*—harsh; rough-surfaced, usually due to roughening or elongation of surface or marginal cells.  
*scales*—the usually small and membranous modified leaves or bracts which subtend the individual flowers or achenes of the Cyperaceae (Fig. 3, C, D, P).  
*sessile*—stalkless, the flower or fruit (or the spikelet) apparently arising directly from the side or apex of the axis on which it is borne.  
*sheath*—the tubular basal portion of the Cyperaceous leaf, the portion which surrounds the culm (Fig. 3, U, X—*sh*).  
*spathiform*—like a spathe (a broad more or less clasping leaf-like structure).  
*species*—a kind of plant; note that *both* the singular and plural form of this word are spelled alike.  
*spike*; *spikelet*—the compact aggregated clusters of Cyperaceous flowers; the term *spike* is usually used to refer to the clusters in *Carex*, while *spikelet* is more generally used in the other genera (although “spikelet” usually means “little spike,” it is seldom used in this sense in the Cyperaceae).  
*staminate flower*—a flower containing only stamens (androecium) and their appendages; small, abortive and nonfunctional pistils are sometimes present (*see also* pistillate flower).  
*stipitate*—with a narrowed, stalk-like base which is still an integral part of the structure under consideration.  
*sub-coriaceous*—somewhat thickened and leathery.  
*suborbicular*—almost round; intermediate between elliptical (Fig. 3, L-5) and orbicular (Fig. 3, L-8).  
*terete*—rounded in cross-sectional shape (Fig. 3, M-4).  
*triangular*—three-cornered or three-angled in cross-sectional shape (Fig. 3, M-3).  
*truncate*—with a squared or cut-off apex (Fig. 3, N-1).  
*tubercle*—the base of the style which is persistent on the apex of the achenes of *Eleocharis*, *Bulbostylis*, *Rhynchospora*, and other genera of the Cyperaceae; this is usually quite different in texture and in color from the achene (Fig. 3, G); compare with *apiculation*.  
*ventral*—the inner (“front” or “upper”) surface of an achene, perigynium, leaf blade, or leaf sheath; the surface which is the nearest to the axis or culm when the structure under consideration is in an upright position (Fig. 3, O, P—*ven*).  
*verrucose*—with a warty surface.  
*wing*—a lateral expansion or extension of the surface of a perigynium (Fig. 3, Q, R) a rachis, a rachilla or a culm.

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## REGISTER OF SCIENTIFIC NAMES

Names of genera are printed in capital letters; species, varieties, etc., are in lower case. Valid species and varieties which occur in Iowa are given in regular type. Synonyms and excluded or doubtfully reported species and varieties are indicated in *italics*. New scientific names proposed in this paper are printed in **bold-face** type.

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# SEED CHARACTERS OF ALFALFA AND CERTAIN OTHER SPECIES OF *MEDICAGO*<sup>1</sup>

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Received July 11, 1946

Considerable difficulty has been experienced from time to time in the Iowa State College Seed Laboratory in accurately determining the presence of bur clover seeds which occasionally occur as incidental admixtures in commercial lots of alfalfa seed. While these seeds have been illustrated in several publications (alfalfa in many), so far as I know, no analysis of the external morphological characters has been made which would indicate whether bur clovers are always distinguishable from alfalfa, and if so, what the most reliable differentiating structures might be. The purpose of this publication is to provide this information. While prepared with the seed analyst primarily in mind, an attempt has been made to make it broad enough so that it may likewise find taxonomic or agronomic application elsewhere. Black medic is also included in order to round out a treatment of the four members of this genus usually encountered. These are all introduced Eurasian species.

The seeds of these plants possess the following general structural characteristics in common: Shape laterally elongate, commonly subreniform to plump-ovoid or irregularly several-sided, angled or "lumpy." Dorsal margin (opposed to hilum side) usually convexly curved. Ventral margin (bearing the hilum) concave, or nearly straight except for a notched or subterminal depression, in side view frequently with a short divergent projection. The configuration of the ventral margin depends, in the main, upon the length of the radicle, which extends along it one-half to three-fourths the length of the seed, and the degree to which the radicle fails to terminate flush with the margin of the cotyledons.

The hilum, a small, circular, generally whitish-scurfy area is located just beyond the termination of the radicle, thus is somewhat distally<sup>3</sup> offset. The nature of the structures or ornamentation adjacent to the hilum varies in different species of *Medicago* and greatly aids in their

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<sup>2</sup>Acknowledgements: The plates were prepared by George Morris. Seeds were made available to the author by a number of individuals, to whom he renders thanks. Particular acknowledgement is due Dr. A. A. Beetle of the California Agricultural Experiment Station who sent to the writer a large selection of seeds of a number of species of *Medicago*. Acknowledgement is also made for helpful suggestions given by several members of the Iowa Laboratory, and by Grady Cope of the Motley Seed Company.

<sup>3</sup>The adjective distal will be used to describe those structures on the short end of the seed at or beyond the hilum; those on the longer edge will be said to be on the radicle or proximal side.

differentiation. A distinct, raised collar or rim is frequently present on the proximal border of the hilum. When well developed, it is semi-circular in form, surrounding the hilum from the proximal and proximal-lateral directions. Seen in lateral view it appears as a short divergent projection just back of the hilum. Distal to the hilum is an area usually marked off from the rest of the seed by distinctive pigmentation. To avoid the use of somewhat ambiguous morphological terms I am going to refer to it merely as the distal ornamentation. This area is commonly elliptic or somewhat wedge-shaped with the narrow end towards the hilum. It initially presents a surface which is dull or somewhat glazed in comparison to the texture of the rest of the seed coat. The terminal portion of this ornamentation, the "distal prominence," is frequently elevated in an umbonate or convex fashion, and commonly possesses a narrow longitudinal slit, the strophiole of some authors. As the seeds become older the pigmentation of this entire area, particularly the prominence, becomes darker; in California bur clover it is soon nearly black. Another dark pigmented area, the "radicle ornamentation" is sometimes observable on the other side of the hilum on the tip of the radicle. These various structures and their positions relative to one another are indicated on the plates.

#### ANALYTICAL KEY

1. Seeds plump-ovoid, mostly 1.3-1.5 times longer than wide; ventral margin neither strongly concave nor notched, sometimes abruptly depressed distal to hilar collar; hilar collar usually strongly developed, evident laterally as a distinct divergent projection.  
*Medicago lupulina*

1. Seeds various in form, commonly subreniform, mostly 1.5-2 times longer than wide; ventral margin commonly concave or notched; hilar collar evident or non-evident.

2. Ventral margin concave or strongly notched; hilar collar strongly developed, arising from base of the notch or concavity; hilum lying partially on base of collar shoulder and in lateral view appearing to slant downwards; dorsal margin of seed strongly convex giving the seeds a distinct curved or falcate appearance; seeds distinctly thin in ventral or dorsal view.  
*M. arabica*

2. Ventral margin various, frequently irregular; hilar collar usually not well developed; hilum horizontal, not appearing to slant; dorsal margin various, usually not as strongly convex as above; seeds usually thicker than above (see plates for these latter comparisons).

3. Distal ornamentation usually dark pigmented, commonly raised terminally and forming an elliptical, convex or umbonate prominence which is visible laterally; seeds relatively regular in appearance, usually straight and plump, scarcely concave ventrally but with a wide conspicuous notch. *M. hispida*

3. Distal ornamentation usually glazed in appearance, scarcely darkened except in old seeds, not raised into a prominence, or if so this structure scarcely discernible laterally; seeds varied in appearance, sub-reniform to irregularly ovoid, frequently angular or twisted, plump or rather thin; ventral margin concave, notched, or distally depressed.  
*M. sativa*

#### DESCRIPTION OF SEEDS

*Medicago arabica* (L.) Huds. Spotted bur clover

Seeds reniform to sub-reniform, straight or slightly twisted longitudinally, distinctly thin in cross section. Dorsal margin strongly curved.

Ventral margin concave or nearly straight, broken by a wide, medial or distally offset notch. Hilar collar arising from center of depression, strongly developed back of hilum, usually about half encircling it but considerably weaker or obscure laterally. Apex of collar nearly on a level with marginal shoulder on each side of concavity. Hilum lying on collar base, somewhat tilted on its inner margin, and slanting downwards. Radicle ornamentation usually not obvious; this area mostly presenting a dull, glazed appearance, sometimes darkened. Distal prominence usually non-evident.

*Medicago hispida* Gaertn. California bur clover

Seeds slightly reniform, quite constant in appearance, averaging larger than the above species. Dorsal margin curved. Ventral aspect nearly straight except for the strong, slightly distally located notch. Seeds plump in cross section. Hilar collar obscure. Hilum basal in notch, horizontal. Radicle ornamentation usually obscure, sometimes darkened and shiny. Distal ornamentation conspicuous, initially glazed and light in color but soon much darkened. Distal prominence strongly developed, evident laterally, frequently conspicuously strophiolate.

*M. hispida*, in its native European home is a wide-spread and polymorphic species. A rather large number of varieties have been described, based principally upon variations in pod structure. I have examined seeds of a number of these forms and find no essential differences from the typical *M. hispida*.

One small lot of seeds inspected, labeled *M. hispida*, bore all the "earmarks" of that species except that the seeds were strongly longitudinally curved. The sample contained no associated burs by which the identification could be verified. Professor Porter of this laboratory informs me that he has on rare occasions encountered such seeds in commercial samples. While it seems likely that these represent some form of *M. hispida*, such disposition must, for the present remain tentative.

*Medicago lupulina* L. Black-medic

Seeds plump-ovoid or compressed-ellipsoidal. Dorsal margin evenly curved. Ventral margin straight, concave or convex, sometimes short concave on each side of collar. Hilar collar usually strongly evident back of the hilum and evident in side view as a distinctive divergent marginal projection, scarcely developed laterally. The ventral margin distal to the collar is sometimes continuous with the radicle portion, but is more commonly depressed. Infrequently the collar is suppressed in which case the ventral margin is abruptly depressed beyond the tip of the radicle to the level of the hilum. Radicle ornamentation frequently evident as a black dot. Distal ornamentation evident or non-evident, usually not striking.

*Medicago sativa* L. Alfalfa

Seeds variously shaped, subreniform to straight, ovoid to variously rounded or angular. Dorsal margin convex, straight or irregular. Ven-



tral margin straight, convex or concave, notched or irregularly distally depressed. Seeds thin or plump in cross section, somewhat twisted or straight longitudinally. Hilar collar non-conspicuous or evident, arising from the base of the ventral concavity or, if the latter is not present, backed up against the radicle tip—when depressed not as high as the shoulders on either side, commonly developed laterally relative to hilum as well as dorsally. Hilum horizontal, not slanted. Radicle ornamentation non-evident or glazed. Distal ornamentation presenting a glazed appearance, in old seeds becoming dark. Distal prominence usually present, glazed or slightly darkened, usually not strongly evident laterally.

#### *Medicago* (other species)

For the sake of completeness it seems desirable to mention other species of *Medicago* occurring in North America. Like the foregoing these all have been introduced from Europe and Asia. *Medicago falcata* L., Yellow-flowered alfalfa, is grown sparingly for experimental purposes. Its seeds are quite similar to those of alfalfa and will be discussed in more detail below relative to hybridization between this species and *M. sativa*. *M. rigidula* (L.) Desr., Tifton bur-clover, has been grown experimentally in Georgia. Seeds of this plant have not been observed. *M. minima* L. occurs in the southeast. Its seeds are similar in general appearance to those of *M. hispida* but considerably smaller. *M. orbicularis* All. is escaped sparingly in California and east to Texas. The seeds are very dissimilar to other species of *Medicago* observed, being strongly compressed and obovate in surface view. *M. apiculata* Willd. is frequently reported from California. It is being considered herein as one of the variants of *M. hispida*.

#### DISCUSSION

California bur clover, spotted bur clover and black medic all possess seeds which are quite constant in appearance and definitely distinctive relative to one another. Alfalfa on the other hand is exceedingly variable, runs a gamut of forms and closely simulates the typical aspect of the seeds of each of the other above mentioned plants. Ovoid alfalfa seeds may resemble those seeds of black medic in which the collar is poorly developed or absent. Usually they may be distinguished by the plump rounded appearance of the black medic and its frequent possession of the small darkened ornamentation area on the radicle. Strongly curved alfalfa seeds with a conspicuous hilar collar in the center of the ventral concavity are very similar to the seeds of spotted bur clover. They usually may be distinguished by the fact that the collar is low in alfalfa (not reaching the shoulder of the concavity) and that the hilum is horizontal and not obliquely slanted. Alfalfa seeds with darkened distal ornamentation, may likewise resemble those of California bur clover. It is doubtful if a conclusive distinction between these two can always be made. The bur clover is ordinarily plumper; the distal prominence is strong and laterally evident, being scarcely noticeable in alfalfa; the bur clover is perhaps a paler lemon-yellow in color and presents a "cleaner" appear-

ance. These are, however, relative conditions and cannot be described in absolute terms; their interpretation rests upon experience with the seeds involved.

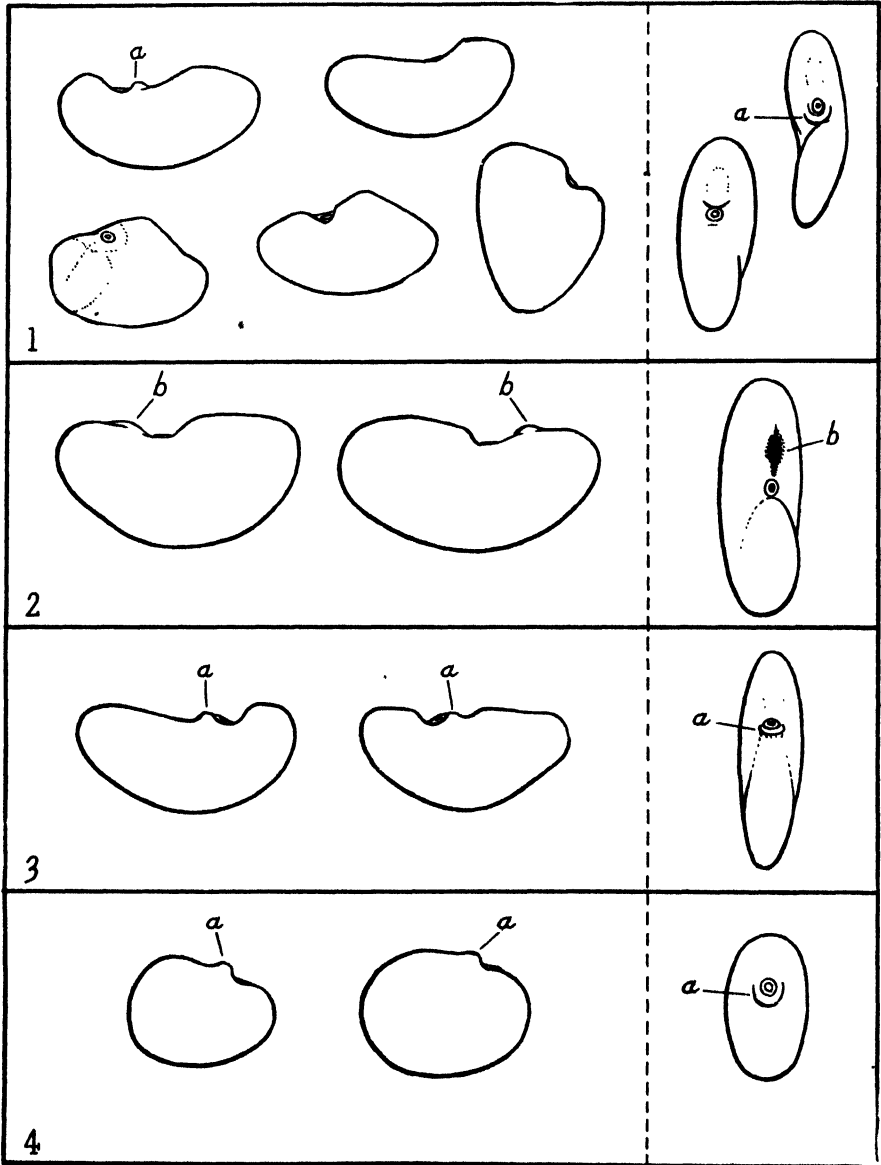
As has been indicated previously bur clover seeds appear to average larger than those of alfalfa. A tabulation of comparative weights of these seeds should be of interest relative to comparison of size. These are as follows:

<i>Species</i>	<i>No. of seeds per gram</i>
Alfalfa .....	575-625
California bur clover .....	350-400
Spotted bur clover .....	525-575
Black medic .....	700-750

The lots of alfalfa and black medic seeds, particularly the latter, which I have had available for inspection appear to be lighter in weight than those used in determining the weights given in the official rules for seed testing (1). The figures in this publication are 586 per gram for black medic and 500 for alfalfa.

The above table is not given for the purpose of indicating that size may be used as a dependable means to differentiate between any of these species of *Medicago*. Size is a notably poor method of distinguishing biological objects unless they are of utterly different stature. For hypothetical purposes let us assume that we have the seeds (or any other structures) of two related plants to be compared, which are similar in every detail except that one averages twice as heavy as the other. The various dimensions of the larger seed, however, will be only 1.26 (cube root of two) times that of the smaller. This difference would in many cases be entirely obscured by the genetic and environmental size variations of the structures involved and no sharp line could be drawn between them. This is the case relative to a comparison between the size of California bur clover and alfalfa, the former of which appears considerably heavier than the latter in the table. The variation in the size of the seeds of alfalfa, like that variation pertaining to morphological appearance, is such that no constant distinction can be made. Spotted bur clover frequently may appear larger than alfalfa, but its similarity in weight is reflected in the thin or compressed cross-section of the seeds.

Perhaps, like myself, the reader may wonder why seed shape is so strikingly variable in alfalfa in comparison to the relative consistency of form displayed by the other species. It should be of interest to note that many of our commercial strains of alfalfa are not true *Medicago sativa* but *M. sativa*-*M. falcata* hybrids. These strains are known as variegated alfalfas, the name reflecting the wide range in flower color exhibited. Grimm, Cossack, Canadian variegated, Ladak, Baltic, and Hardigan are all listed by Westover (6) as belonging to this group. Just as the flower color in these hybrid forms has displayed a great diversity, so perhaps recombination and segregation of various complimentary factors for seed shape has, in part, caused the observed diversity of alfalfa seeds. Seeds of *M. falcata* are not strikingly different from those of



(1) Alfalfa, *Medicago sativa*; (2) California bur clover, *Medicago hispida*; (3) Spotted bur clover, *Medicago arabica*; (4) Black medic, *Medicago lupulina*.  
(a. Hilum; b. Distal prominence)

alfalfa, except that, in the few available for inspection, none of the sub-reniform type common in alfalfa, was seen. Oakley and Garver (4) describe these seeds as follows, "In general appearance the seeds closely resemble those of *Medicago sativa*, but a careful examination shows them to be appreciably smaller, and decidedly more angular. The radicle is also more prominent and in some seeds the hilum is very marked while in others it is scarcely apparent. When examined under the lens the seeds show a slightly roughened surface."

The pods of these four species of *Medicago* are quite distinctive. That of Black medic differs from the others in being one-seeded and only curved while the others are several seeded and spirally twisted. Those of the bur clovers are brownish and possess divergent spine-like projections, while alfalfa pods are black in color and spineless. Pods of California bur clover are loosely twisted, making only about two-three complete revolutions, while those of spotted bur clover are much thinner, more tightly spiralled, making three-four revolutions.

A few facts about the distribution of these plants may be pertinent. Black medic probably occurs the country over and may be occasionally expected in alfalfa as an incidental seed from any source in the United States. The bur clovers are more limited in their distribution. *Medicago arabica* is most abundant in the southeast but also occurs in California. *M. hispida* is common west of the Cascades, both cultivated and as an escape, and is also found to a lesser degree across the southern part of the country to the east (McKee, 3; Jepson, 2; Small, 5). These two species are only occasional throughout the rest of the country and thus scarcely overlap the important alfalfa seed producing centers (except for that in southwestern Arizona) which are mostly more northerly in semi-arid regions of the midwest and west. Thus bur clover is not to be expected as an abundant contaminant in alfalfa from the majority of sources in the country, but its occasional incidental appearance and the fact that it has at times been employed as an adulterant for alfalfa indicates that it should always be kept in mind by the analyst.

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# THE TYPES OF AUCHENORRHYNCHOUS HOMOPTERA IN THE IOWA STATE COLLEGE COLLECTION<sup>1</sup>

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*From the Entomology and Economic Zoology Section, Iowa Agricultural Experiment Station.*

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The early taxonomic work of E. P. Van Duzee, Herbert Osborn, and E. D. Ball marked the beginning of comprehensive studies of the auchenorrhynchous Homoptera of eastern and central North America. The basic nature of these studies, which resulted in the description of many species and the publication of several revisional papers, makes the material upon which they were based of more than usual interest. For the most part the identity of the various species described by these authors is well known, but in a few instances, involving closely related forms, some confusion exists as to the proper application of names. In order to establish a sound basis for future work, it seems desirable to select a lectotype for those species of which the original descriptions were based upon more than a single specimen. In the present paper I have attempted to do this for all species the types of which might be presumed to be in the Iowa State College collection, unless earlier lectotype selections have been made and published.

Type material of most of the species described by Van Duzee prior to 1898, and by Osborn or Osborn and Ball prior to 1899, is now in the Iowa State College collection. These collections were first arranged and placed in insect boxes by Osborn and Ball. Later the specimens were transferred to wooden blocks in glass-top cases by assistants under the direction of Prof. H. E. Summers, formerly head of the Department of Zoology and Entomology. It was apparently during this transfer and the subsequent use of the collections in class work that many of the identification labels attached by the original authors were removed and discarded. The collections are now well housed in unit trays in Comstock drawers contained in steel cabinets.

At the time of its acquisition by the College, about 1898, the Van Duzee collection presumably contained nearly all the type material upon which his species were based, excepting certain specimens borrowed from institutions and returned to those repositories and a limited amount of material distributed to a few institutions by Van Duzee himself. On the other hand, the Osborn and Ball material, when adequate, was habitually divided into three sets, one set going to each of the two authors

<sup>1</sup> Bureau of Entomology and Plant Quarantine Manuscript No. 8252, Agricultural Research Administration, United States Department of Agriculture.

<sup>2</sup> Resident collaborator, Entomology and Economic Zoology Section, Iowa Agricultural Experiment Station, while making a study of the types of Homoptera in the insect collection of Iowa State College.

and one to the Iowa State College. In many instances a fourth set was segregated and sent to the United States National Museum. A further distribution of material has resulted from exchanges and gifts, and no attempt is made to determine the present location of all syntypes.

The arrangement of the Osborn, Osborn and Ball, and Van Duzee species here treated is an alphabetical one under each family, since such an arrangement seems most convenient for users of the paper. The name under which the species was originally described is given, if it differs from the currently accepted name which is used in the alphabetical arrangement, and is followed by a bibliographic citation to the original description. The originally published statement concerning material upon which a given species was based is quoted; if additional essential information is available from some other part of the same paper, such information is included but is enclosed in brackets. Following this quotation is a list of the syntypes now in the Iowa State College collection, together with their pin-label data and other pertinent information. Determination labels attached to syntypes are in the handwriting of the author of the species unless otherwise indicated. Next is a statement of the lectotype selection if such a selection is made. Synonyms are cross-referenced. An alphabetical index of specific names under each family, showing the present generic placement of each species, is included. In this index generic names enclosed in brackets are the names with which the specific names were originally combined if the generic placement has been changed.

It has not always been possible to indicate the collector of type material discussed. In this connection it should be noted that a great deal of the Iowa material labeled with Experiment Station numbers was collected or reared by E. D. Ball, and that practically all such material collected in 1896 and 1897 should be credited to Ball.

In listing pin label data I have omitted, for purposes of brevity, reference to red "cotype" labels bearing the name of the species and its author. These were attached to nearly all material in 1936, under the supervision of Dr. Herbert Osborn, and their presence on all syntypes excepting the Fulgoroidea may be assumed unless otherwise indicated. Lectotypes which I have selected during the course of this work may be identified by a yellow pin label bearing the name under which the species was originally described, and the indication "LECTOTYPE" and "Oman 1946."

In this work I have followed Frizzel (Amer. Midland Nat. 14: 637-668, 1933) in terminology of types, using "syntype" in preference to "cotype" to refer to any specimen of the author's original material when no holotype was designated, and "lectotype" as a syntype chosen subsequent to the original description to take the place which in other cases a holotype occupies. In the event that a species was described from a single specimen no selection is required, that specimen being, by definition, a holotype. Allotypes are not designated.

At present there is not necessarily the assurance that the International Commission on Zoological Nomenclature will recognize the selection

and designation of specimens by subsequent workers as a basis for species fixation, but the general tendency of workers has been to accept such selections, and it seems safe to assume that the interpretation of species can be so stabilized whenever confusion exists.

The preparation of this paper has been facilitated by a manuscript on the same subject prepared by Herbert Osborn and Dale R. Lindsay, and by the helpful cooperation of Carl J. Drake who has placed at my disposal adequate clerical and other assistance. Figures 2, 4, 5, 13 A-H, and 14 have been prepared by Sue Sparks Lewis, the remainder by myself.

For the convenience of other workers there is appended an alphabetical list, by families, of other types of auchenorrhynchos Homoptera in the Iowa State College collection.

#### FAMILY CERCOPIDAE

##### *Aphrophora annulata* Ball

Iowa Acad. Sci. Proc. 6:216, 1899.

"Described from sixteen examples labeled 'Wasatch, Utah, 6-27-91' received through the kindness of Mr. Otto Heidemann."

1 ♂ — "Wasatch, Ut 27-6-91, Type."

The above-indicated specimen is a syntype.

Lectotype male, "Wasatch, Ut 27-6-91, Type," from the E. D. Ball material in the United States National Museum, here designated.

##### *Aphrophora irrorata* Ball

Iowa Acad. Sci. Proc. 6:214, 1899.

"Described from several examples received from Professor Bruner, taken in Sioux county, Nebraska (War-Bonnet Cañon), and others taken in Rist Cañon (Ft. Collins), Colo."

1 ♂ — "Squaw Canon, Sioux Co., Neb. July 22, 1892, Type."

1 ♀ — "Squaw Canon, Sioux Co., Neb. July 18, 1892, Type."

The above indicated specimens are syntypes.

Lectotype male, "Squaw Canon, Sioux Co., Neb. July 20, 1892, Type," from the E. D. Ball material in the United States National Museum, here designated.

Although the pin label data of the above indicated specimens do not agree exactly with Ball's original locality records, there is no doubt that all are syntypes.

#### FAMILY MEMBRACIDAE

##### *Glossonotus godingi* (Van D.)

*Thelia godingi* Van Duzee, Ent. News 6: 203, 1895.

". . . I have taken a number of individuals about Buffalo, mostly on bushes of wild black cherry in June and July."

1 ♂, 1 ♀ — "Buffalo, N. Y. July 1886, E. P. V. Coll., Type."

2 ♀ ♀ — "Buffalo, N. Y. June 1887, E. P. V. Coll."

Lectotype male, the above indicated specimen, here designated.



## FAMILY CICADELLIDAE

*Aceratagallia cinerea* (O. and B.)

*Agallia cinerea* Osborn and Ball, Davenport Acad. Nat. Sci. Proc. 7:62, 1898.

"Described from numerous examples from Iowa and Colorado.

"The Iowa examples were taken at Little Rock and Sioux City in July, from high gravelly points where plants characteristic of the plain region, such as *Bouteloas* and *Artemisias* predominated, and from which several other species of western Hemiptera were taken. The Colorado specimens were received through the kindness of Prof. Gillette."

3 ♂♂, 1 ♀—"Colo. 2279, Type."

3 ♂♂, 4 ♀♀—"Colo. 2282, Type."

1 ♀—"Colo. 290, Type, 125," is *humilis* Oman.

Lectotype male, "Colo. 2279, Type," here designated.

Oman (U. S. D. A. Tech. Bul. 372:55, 1933) discussed the identity of *cinerea*, the type series of which included specimens of *vulgaris* Oman in addition to the material listed above.

*Aceratagallia gillettei* (O. and B.)

*Agallia gillettei* Osborn and Ball, Davenport Acad. Nat. Sci. Proc. 7:60, 1898.

"Described from numerous examples collected in Arizona by Prof. Gillette, . . ."

1 ♂, 1 ♀—"Ariz. 2102, Gillette, Type."

2 ♂♂, 1 ♀—"Ariz. 2217, Type."

2 ♂♂, 2 ♀♀—"Ariz. 2072, Gillette, Type."

1 ♀—"Ariz. 2089, Gillette, Type."

Lectotype male, "Ariz. 2102, Gillette, type," here designated.

*Aceratagallia longula* (Van D.)

*Agallia longula* Van Duzee, Canad. Ent. 26:92, 1894.

*Agallia lyrata* Baker, Psyche 8:199, 1898. New synonymy.

Van Duzee's *longula* was based upon "two highly-coloured examples from California . . ."

No syntypes of *longula* found in the Iowa State College collection.

Lectotype male, "Los Angel, PRUhler Collection," and bearing the name label "*Agallia longula* Uhl. Cal." in Uhler's handwriting, here designated, in the United States National Museum collection.

The above designated lectotype, one of three specimens bearing identical pin label data, is believed to be one of the two examples mentioned by Van Duzee in connection with his description of *uhleri*. This assumption is strengthened by Van Duzee's statement that the two specimens "... came with the name *Agallia longula*, Uhl.," indicating material borrowed and probably returned to the sender.

At the time I prepared my revision of North American agallian leafhoppers (U. S. D. A. Tech. Bul. 372, 93 pp., 1933) I was not aware of the status of manuscript names quoted in synonymy. If the above selection of a lectotype is from the proper specimens, as I believe it to be, it is apparent that the California records of *uhleri* refer to the species

formerly known as *lyrata* (Baker) rather than to *curvata* Oman, as indicated on page 63 of the above cited publication.

*Aceratagallia uhleri* (Van D.)

*Agallia uhleri* Van Duzee, Canad. Ent. 26:91, 1894.

*Agallia enervis* Van Duzee, Canad. Ent. 26:92, 1894.

*Agallia venata* Van Duzee, Canad. Ent. 26:92, 1894.

"Colorado, Arizona, California. Described from ten examples representing both sexes. This plain little insect I have received from several correspondents labeled *Agallia venata*, Uhl., and *Agallia enervis*, Uhl., and two highly-coloured examples from California came with the name *Agallia longula*, Uhl. The California material was received from Mr. Coquillett; those from Arizona were from the Morrison collection, and the specimens from Colorado I owe to the kindness of Prof. C. P. Gillette."

1 ♀—"Arizona, C. U. Lot 34, Cornell U. Lot 45, Sub. 423, Type *Agallia enervis* Uhl."

1 ♀—"Arizona, C. U. Lot 34, Cornell U. Lot 45, Sub. 424, *Agallia venata* Uhl."

1 ♂, 1 ♀—"Arizona, C. U. Lot 34, Cornell U. Lot 45, Sub. 424, Type, *Agallia uhleri* Van D.," mounted on one pin.

1 ♀—"Arizona, C. U. Lot 34, Cornell U. Lot 45, Sub. 423, Type" belongs to the *sanguinolenta* complex.

Lectotype female, "Arizona, C. U. Lot 34, Cornell U. Lot 45, Sub. 423, Type, *Agallia enervis* Uhl." is here designated for *uhleri* and *enervis*.

Lectotype female, "Arizona, C. U. Lot 34, Cornell U. Lot 45, Sub. 424, *Agallia venata* Uhl." is here designated for *venata*.

Van Duzee's publication of the specific names *enervis*, *longula*, and *venata* validated them for future use. Designation of lectotypes of *enervis* and *venata*, here made, fixes them as synonyms of *uhleri*.

Colorado specimens at hand, consisting of four females labeled "Colo. 516," "Colo. 2143," "Colo. 2180," and "Colo. 2294" are probably syntypes, but not definitely identifiable as such.

*Acinopterus acuminatus* Van D.

Psyche 6:308, 1892.

"Described from 5 ♂, 3 ♀. Maryland, Sept. 29th and Aug. 4th on pines (Uhler). N. Carolina (Osborn). New Jersey (Uhler). Mountains of N. W. Colorado (Gillette). California (Coquillett)."

No syntypes found.

*Agallia constricta* Van D.

Canad. Ent. 26:90, 1894.

"New Jersey, Mississippi, Florida. Described from numerous examples received from Mr. Howard Evarts Weed, Prof. J. B. Smith, and others."

3 ♂♂, 2 ♀♀—"Miss., Type," one female with "*Agallia constricta* Van D."

1 ♂—"Fla., Type."

1 ♀—"N. J., Smith, Type."

Lectotype male, "Miss., Type," here designated.

One male and one female, labeled "cotype, *Agallia constricta*" bear pin label data "Ag. Coll., Miss. Jl. '94, H. E. Weed" and cannot be syntypes since the species was described in April 1894.

*Agallia enervis* Van Duzee, 1894 = *Aceratagallia uhleri* (Van D., 1894).

*Agallia modesta* O. and B.

Davenport Acad. Nat. Sci. Proc. 7:51, 1898.

"Described from seventeen examples collected in Mexico (Osborn)."

3 ♂♂, 1 ♀—"Minatitlan, Mex. Feb. 1, '92, H. Osborn, Collector, Type."

1 ♂, 1 ♀—"Motzorongo, V. C., Mex. Jan. '92, H. Osborn, Collector, Type."

1 ♀—"Orizaba, V. C., Mex. Jan. 9-16, '92, H. Osborn, Collector, Type," is not *modesta*.

Lectotype male, "Minatitlan, Mex. Feb. 1, '92, H. Osborn, Collector, Type," here designated.

*Agallia producta* O. and B.

Davenport Acad. Nat. Sci. Proc. 7:52, 1898.

"Described from ten examples collected January 12, at Orizaba, V. C., Mexico (Osborn)."

2 ♂♂, 2 ♀♀—"Orizaba, V. C., Mex. Jan. 9-16, '92, H. Osborn, Collector, Type," one male without "cotype, *Agallia producta*" label.

1 ♂—"Orizaba, V. C., Mex. Jan. 9-16, '92, H. Osborn, Collector, Type" is not *producta*.

Lectotype male, "Orizaba, V. C., Mex. Jan. 9-16, '92, H. Osborn, Collector, Type," here designated.

*Agallia quadripunctata* (Prov.)

*Bythoscopus quadripunctatus* Provancher, Nat. Canad. 4:376, 1872.

*Ulopa canadensis* Van Duzee, Amer. Ent. Soc. Trans. 19:301, 1892. [Nymphs.]

Van Duzee's *canadensis* was from "Canada. Described from six male examples, which, with other specimens of the same species, were received from Mr. Alva H. Kilman, of Ridgeway, Welland County, and Mr. W. Hague Harrington, of Ottawa."

3 nymphs—"Ridgeway, Ont., Kilman Coll., Type," on one pin bearing the determination label "*Ulopa canadensis* Van D."

1 nymph—"W. H. H., Ottawa, Can., Type."

Lectotype, "Ridgeway, Ont., Kilman Coll., Type," lowermost specimen on pin, here designated for *canadensis*.

*Agallia venata* Van Duzee, 1894 = *Aceratagallia uhleri* (Van D., 1894).

*Agalliopsis oculata* (Van D.)

*Agallia oculata* Van Duzee, Ent. Amer. 6:38, 1890.

[California, coll. Coquillett.] "Described from two individuals, representing both sexes (No. 278)."

1 ♀—"California, Coquillett, Type" and with determination label "*Agallia oculata* Van Duzee."

Lectotype female indicated above, here designated.

*Agalliopsis tenella* (O. and B.)

*Agallia tenella* Osborn and Ball, Davenport Acad. Nat. Sci. Proc. 7:56, 1898.

"Described from three females, Vera Cruz, Mex. (Osborn)."

1 ♀—"Orizaba, V. C., Mex. Jan. 9-16, '92, H. Osborn Collector, Type."

2 ♀ ♀—"Cordova, V. C., Mex. Jan. 23, '92, H. Osborn Collector, Type," are not *tenella*.

Lectotype female, "Orizaba, V. C., Mex. Jan. 9-16, '92, H. Osborn collector, Type," here designated.

Oman (U. S. D. A. Tech. Bul. 372:18-19, 1933) has discussed the differentiation of the species confused in the type series.

*Aligia inscripta* (Van D.)

*Allygus inscriptus* Van Duzee, Ent. Amer. 6:92, 1890.

[California, coll. Coquillett.] "Described from two males (No. 222)."

1 ♂—"California, Coquillett, Type," with genital capsule removed, dissected, and contained in a small vial attached to the pin.

Lectotype male, the above indicated specimen, here designated.

*Aligia modesta* (O. and B.)

*Eutettix modesta* Osborn and Ball, Davenport Acad. Nat. Sci. Proc. 7:98, 1898.

"Described from one male and one female swept from second growth oaks, Ames, Iowa."

1 ♂—"Ames, Ia. 7-29-91 Exp. Sta., Type," bearing a label "ALLO-TYPE *Eutettix modesta* O. and B.," and with genital capsule removed, dissected, and contained in a small vial attached to the pin.

Lectotype male, the above indicated specimen, here designated.

*Amplicephalus concentricus* (Van D.)

(Figs. 1, A, B)

*Deltocephalus concentricus* Van Duzee, Buffalo Soc. Nat. Sci. Bul. 5:208, 1894.

"Mountains of N. W. Colorado. Described from a single male example received from Prof. C. P. Gillette."

1 ♂—"Col. 158, Type" and with determination label "*Deltocephalus concentricus* Van Duzee." Genital capsule cleared and contained in small vial attached to pin.

Holotype male, the above indicated specimen.

*Amplicephalus osborni* (Van D.)

*Deltocephalus osborni* Van Duzee, Amer. Ent. Soc. Trans. 19:304, 1892.

"New York and Iowa. Described from one male and four female examples taken at Lancaster, N. Y., Sept. 3, 1888. These were swept from grass and weeds near the borders of a low swampy wood. I have also had the pleasure of examining an Iowa specimen received from Prof. Herbert Osborn, . . ."

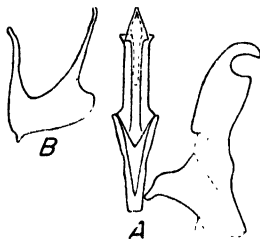


FIG. 1. *Amplicephalus concentricus* (Van Duzee). Internal male genitalia. A, style, connective, and aedeagus, ventral view; B, aedeagus, lateral view.

1 ♂, 2 ♀ ♀—"Lancaster, N. Y. 9-3-88, E. P. V. Coll., Type," the male and one female on a single pin bearing the determination label "*Deltocephalus Osborni* Van D."

Lectotype male, the above indicated specimen, here designated.

*Amplicephalus simplarius* (O. and B.)

*Deltocephalus simplex* Van Duzee, Amer. Ent. Soc. Trans. 19:304, 1892.

*Athysanus simplarius* Osborn and Ball, Ohio Naturalist 2:249, 1902. New name for *Deltocephalus simplex* Van Duzee, 1892, a homonym of *Jassus simplex* Herrich-Schaeffer, 1834, by its being referred to *Athysanus* by Osborn and Ball, l. c.

"Described from one male and four female examples. Canton Marsh, Md. October 2nd, Mr. Uhler. Astoria, L. I., July, and Hoboken, N. J., June, Mr. E. B. Southwick."

1 ♂—"E. B. Southwick [on green paper], Type," and with three small, green paper tabs attached.

1 ♀—"E. B. Southwick, Type," and with one purple paper tab attached.

1 ♀—"Canton Marsh, Oct. 2, Type."

Lectotype male, the above indicated specimen, here designated.

*Atanus perspicillatus* (O. and B.)

(Fig. 2)

*Thamnotettix perspicillata* Osborn and Ball, Iowa Acad. Sci. Proc. 4:227, 1897.

"Described from two females and four males, Ames, Iowa."

1 ♂—"Ames, Ia. 7-11-96, Type."

1 ♂—"Ames, Ia. 7-16-96, Exp. Sta. AC 2841, Type," with nymphal skin attached to tip of abdomen.

Lectotype male, "Ames, Ia. 7-11-96, Type," here designated.

*Balclutha abdominalis* (Van D.)

*Gnathodus abdominalis* Van Duzee, Canad. Ent. 24:113, 1892.

"New Jersey. Described from two male examples received from Prof. Smith, and taken at New Brunswick, July 20th, and Jamesburgh, July 15th."

1 ♂—"Jamesburgh, N. J., 7. 15, Type," with red label "Holotype, *Gnathodus abdominalis* Van D." and determination label "*Balclutha abdominalis* V. D. Type," in DeLong's (?) handwriting. Genital structures dissected and contained in small vial attached to pin.

Lectotype male, the above indicated specimen, here designated.

A female specimen "N. Brunsw., N. J. 7-20" and with a "cotype, *Gnathodus abdominalis*" label attached, is not considered a syntype be-

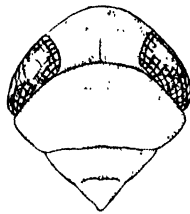


FIG. 2. *Atanus perspicillatus* (O. and B.). Head and thorax, dorsal view.

cause it seems unlikely that Van Duzee would mistake the sex of a specimen in this group. Furthermore, this specimen lacks the small "Type" label usually attached to Van Duzee's syntypes.

*Balclutha impicta* (Van D.)

*Gnathodus impictus* Van Duzee, Canad. Ent. 24:113, 1892.

"New Jersey. Described from a single pair received from Prof. J. B. Smith, and labeled 'New Brunswick, July 20.'"

1 ♂—"N. Brunsw., N. J., 7-20, Type," with red label "Lectotype, *Balclutha impictus* Van D."

Lectotype male, the above indicated specimen, here designated.

Five topotype specimens, bearing data identical with that of the lectotype, mounted on a single pin with a "Cotype, *Balclutha impictus* Van D." label attached, are eliminated from consideration as syntypes because of the restriction on the number of specimens in the original type series.

*Ballana atridorsum* (Van. D.)

*Thamnotettix atridorsum* Van Duzee, Canad. Ent. 26:92, 1894.

"Colorado. Described from three female specimens received from Prof. C. P. Gillette."

No syntypes of this species found in Iowa State College collection. One pin with label "Colo. 633, Type" and attached note "Specimen lost—See H. O. note."

In the United States National Museum collection is a female specimen which fits Van Duzee's description of *atridorsum* well and is believed to be a syntype. It bears the following data: "Colo. 1039, Type," with red name label "*Thamnotettix atridorsum* Van D." in C. F. Baker's handwriting.

Lectotype female, the above indicated specimen, here designated.

The above designated lectotype does not represent the same species as does the neotype selected by DeLong (Ohio Jour. Sci. 37:105, 1937), but is smaller and closely related to *Ballana transea* DeLong, 1937. Study of much additional material of both sexes is needed to clarify the identity of *atridorsum* Van Duzee.

*Bandara johnsoni* (Van D.)

(Figs. 3, A, B)

*Euttettix johnsoni* Van Duzee, Canad. Ent. 26:137, 1894.

"Described from one male and two female examples taken at Philadelphia, Pa., by Mr. C. W. Johnson."

1 ♂, 1 ♀—"Phila., Pa., Type," the male with determination label

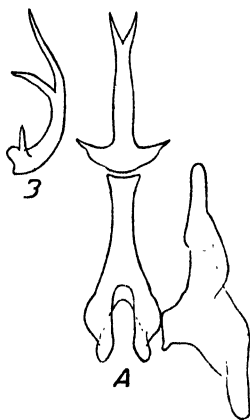


FIG. 3. *Bandara johnsoni* (Van Duzee). Internal male genitalia. A, style, connective, and aedeagus, ventral view; B, aedeagus, lateral view.

"*Eutettix Johnsoni* Van D." Genital capsule cleared and contained in small vial attached to pin.

Lectotype male, the above indicated specimen, here designated.

*Bythoscopus distinctus* Van Duzee, 1890 = *Oncopsis verticis* (Say, 1831).

*Carneocephala floridana* (Ball)

*Draeculacephala floridana* Ball, Iowa Acad. Sci. Proc. 8:72, 1901.

"Described from two males from Charlotte Harbor, Florida, from the Iowa State College collection (Van D. Coll.) through the kindness of Professor Summers."

1 ♂ —“Ch. Hbr., Fla., 2, Type” and bearing the determination label in Ball’s handwriting “*Draeculacephala floridana* Ball, Type.”

Lectotype male, the above indicated specimen, here designated.

*Carneocephala gillettei* (Ball)

*Draeculacephala gillettei* Ball, Iowa Acad. Sci. Proc. 8:72, 1901.

“Described from eighteen specimens from La Salle and Fort Collins, Colo. The first specimens were collected by Professor Gillette.”

1 ♂, 1 ♀ —“N. Colo., 3-26-98, Type.”

The above specimens are syntypes.

Lectotype male, “N. Colo., 3-26-98, Type,” from the Ball collection in the United States National Museum, here designated.

*Chlorotettix balli* Osb.

Osborn, Iowa Acad. Sci. Proc. 5:246, 1898.

“Described from eleven females and four males, collected at Ames, Iowa, July 4th-11th-29th, and August 3, 1896.”

1 ♂ —“Ames, Ia. 7-24-96, Exp. Sta., Type.”

1 ♀ —“Ames, Ia. 8-3-96, Exp. Sta., Type.”

4 ♀ ♀ —“Ames, Ia. 7-29-96, Exp. Sta., Type.”

1 ♀ —“Ames, Ia. 7-2-91, Exp. Sta., Type.”

1 ♀ —“Ames, Ia., Type.”

Lectotype male, the above indicated specimen, here designated.

*Chlorotettix galbanatus* Van D.

Psyche 6:310, 1892.

“Described from three examples received from Mr. E. B. Southwick and captured by him in the vicinity of New York City in June and July; and one specimen taken by Mr. W. J. Palmer, Jr., of this city, on Mt. Balsam, near Asheville, N. C., in July, 1889.”

1 ♀ —“E. B. Southwick [on green paper], Type” and pin bearing a gray paper tab.

1 ♀ —“Type.”

1 ♀ —“Balsam, N. C., 7-23-90, W. J. P. Coll., Type,” is *Doleranus vividus* (Crumb).

Lectotype female, “E. B. Southwick, Type,” here designated.

A male specimen labeled “New York, N. Y. 8-28-188[3?] N C, E. B. Southwick” without the characteristic “Type” label but with a red “Cotype” label, is not considered a syntype.

*Chlorotettix lusorius* (O. and B.)

*Thamnotettix lusoria* Osborn and Ball, Iowa Acad. Sci. Proc. 4:226, 1897.

“Described from eight males and ten females all collected at Ames, Iowa.”

1 ♂ —“Ames, Ia. 7-16-96, Type.”

2 ♂ ♂ —“Ames, Ia. 6-17-96, Type.”



1 ♂ —“Ames, Ia. 7-2-96, Type.”

1 ♀ —“Ames, Ia. 6-24-96, Type.”

1 ♀ —“Ames, Ia. 7-4-96, Type.”

1 ♀ —“Ames, Ia. 6-28-96, Type.”

Lectotype male, “Ames, Ia. 7-16-96, Type,” here designated.

*Chlorotettix necopinus* Van D.

*Chlorotettix necopina* Van Duzee, Canad. Ent. 25:282, 1893.

“Mississippi. Described from two female examples kindly sent me by Mr. Howard Evarts Weed.”

1 ♀ —“Miss., Type.”

Lectotype female, the above indicated specimen, here designated.

*Chlorotettix spatulatus* O. and B.

*Chlorotettix spatulata* Osborn and Ball, Iowa Acad. Sci. Proc. 4:225, 1897.

“Described from forty-two examples collected at Ames, Iowa. It has also been received from Colorado (Gillette) and Nebraska (Bruner).”

1 ♂ —“Ames, Ia. 8-15-96, Type.”

1 ♂ —“Ames, Ia. 8-11-96, Type.”

1 ♂ —“Ames, Ia. 9-22-96, Type.”

1 ♀ —“Ames, Ia. 5-29-96, Type.”

1 ♀ —“Ames, Ia. 6-15-96, Type.”

1 ♀ —“Ames, Ia. 9-11-96, Type.”

1 ♀ —“Ames, Ia. 9-3-96, Type.”

1 ♀ —“Iowa, Type.”

1 ♀ —“Ames, Ia. 6-13-96, Osborn.”

Lectotype male, “Ames, Ia. 8-15-96, Type,” here designated.

*Chlorotettix viridius* Van D.

*Psyche* 6:309, 1892.

“Described from six examples collected near New York City by Mr. E. B. Southwick, in July; one female taken at New Brunswick, N. J., July 20th, by Prof. J. B. Smith, and numerous specimens received from Mr. Howard Evarts Weed, taken in Mississippi.”

1 ♂ —“Miss., Type,” and with determination label “*Chlorotettix viridius* V D.”

1 ♂ —“Miss., Type.”

1 ♂ —“Type,” and pin bearing a purple paper tab.

2 ♀ ♀ —“Miss., Type.”

1 ♀ —“E. B. Southwick [on green paper], Type.”

Lectotype male, “Miss., Type, *Chlorotettix viridis* V D.,” here designated.

*Cicadula ciliata* (Osb.)

*Thamnotettix ciliata* Osborn, Iowa Acad. Sci. Proc. 5:244, 1898.

“Described from numerous examples of both sexes collected in Iowa and one female from Colorado (Gillette). Adults at Ames from June 2d

to July 2d, and from August 27th to October 9th. Three specimens from Little Rock, July 2d (Ball) and two from Algona, Iowa, May 9th (Mally)."

1 ♂—"Ames, Ia. 9-25-96, Exp. Sta., Type."

1 ♂, 1 ♀—"Ames, Ia. 9-22-96, Exp. Sta., Type."

1 ♂—"Ames, Ia. 8-20-97, Type."

1 ♂, 1 ♀—"Ames, Ia. 10-5-96, Exp. Sta., Type."

1 ♀—"Ames, Ia. 10-13-96, Exp. Sta., Type."

Lectotype male, "Ames, Ia. 9-25-96, Exp. Sta., Type," here designated.

*Cicadula cyperacea* (Osb.)

*Thamnotettix cyperaceus* Osborn, Iowa Acad. Sci. Proc. 5:245, 1898.

"Described from four males and four females collected from *Carex* at Ames, Iowa, October 6, 1897."

2 ♂, 2 ♀—"Exp. Sta., Oct. 6, '97, Ames, Ia., Type."

1 ♂, 1 ♀—"Exp. Sta., Sept. 10, '97, Ames, Ia., Type."

1 ♀—"Exp. Sta., Oct. 9, '97, Ames, Ia., Type."

Lectotype male, "Exp. Sta., Oct. 6, '97, Ames, Ia., Type," here designated.

*Cicadula longiseta* (Van D.)

*Thamnotettix longiseta* Van Duzee, Canad. Ent. 24:266, 1892.

"Northwestern Colorado. Described from a single female example received from Prof. C. P. Gillette."

1 ♀—"Col., Ac. Cat. 169, 42, Type."

Holotype female, the above indicated specimen.

*Cicadula punctifrons* var. *americana* Van Duzee, 1891 = *Davisonia punctifrons* var. *repleta* (Fieb., 1885).

*Cicadula smithi* (Van D.)

*Thamnotettix Smithi* Van Duzee, Canad. Ent. 24:266, 1892.

"New Brunswick, N. J. Described from a single male example kindly given me by its captor, Prof. J. B. Smith. . . ."

1 ♂—"N. Brunsw., N. J., 24, Type."

Holotype male, the above indicated specimen.

*Ciminius hartii* (Ball)

*Tettigonia hartii* Ball, Iowa Acad. Sci. Proc. 8:61, 1901.

"Specimens are at hand from southern Ohio, a pair each from southern Illinois, Florida and Mississippi, and several females from Texas, New Mexico and Cuba."

1 ♂—"Miss., 16, Type."

1 ♀—"Florida, Slosson, Type," with determination label "*Tettigonia hartii* Ball, Types."

2 ♀—"Cuba, Combs, Type," one with determination label "*Tettigonia hartii* EDB."

1 ♂, 3 ♀—"Albuq., N. M., Type," one female with determination label "*Tettigonia hartii* EDB."

The Cuba and New Mexico specimens probably represent two species, each distinct from *hartii*.

The above indicated specimens are syntypes.

Lectotype female, "Miss., 39, Type," in E. D. Ball collection, United States National Museum, here designated.

*Cloanthanus cinereus* (O. and B.)

*Platymetopius cinereus* Osborn and Ball, Iowa. Acad. Sci. Proc. 4:193, 1897.

[Iowa.] ". . . the most abundant species at Ames, occurring everywhere that wild grasses are found. Specimens have also been received from Kansas, Nebraska, and Arizona, . . ."

1 ♂ — "Ames, Ia., Exp. Sta. AC 1796, Type."

1 ♂ — "Ames, Ia. 5-26-96, Type."

1 ♂ — "Ames, Ia. 8-4-96, Type."

1 ♀ — "Ames, Ia. 5-29-96, Type."

2 ♀ ♀ — "Ames, Ia. 6-4-96, Type."

Lectotype male, "Ames, Ia., Exp. Sta. AC 1796, Type," here designated.

*Cloanthanus frontalis* (Van D.)

*Platymetopius frontalis* Van Duzee, Canad. Ent. 22:112, 1890.

"Described from two ♂ and five ♀ examples. Buffalo, N. Y., June and September; Ames, Iowa, (H. Osborn)."

1 ♂, 2 ♀ ♀ — "Buffalo, N. Y., June 1887, E. P. V. Coll., Type," mounted on one pin.

1 ♀ — "Buffalo, N. Y., Sept. 1886, E. P. V. Coll., Type."

Lectotype male, the above indicated specimen, uppermost on the pin, here designated.

*Cloanthanus fuscifrons* (Van D.)

*Platymetopius fuscifrons* Van Duzee, Buffalo Soc. Nat. Sci. Bul. 5:206, 1894.

"Arizona. Described from one male and two female examples received from the Morrison Collection at Cornell University."

1 ♂, 1 ♀ — "Arizona, C. U. Lot 34, Cornell U. Lot 45, Sub. 457, Type."

Lectotype male, the above indicated specimen, here designated.

*Cloanthanus loricatus* (Van D.)

*Platymetopius loricatus* Van Duzee, Buffalo Soc. Nat. Sci. Bul. 5:205, 1894.

"California. Described from four male examples received from Mr. D. W. Coquillett."

2 ♂ ♂ — "California, Coquillett, Type."

Lectotype male, one of the above indicated specimens, here designated.

*Cochlorhinus unispinosus* Beamer

*Deltocephalus coquilletti* Van Duzee, Ent. Amer. 6:95, 1890.

*Cochlorhinus unispinosus* Beamer, Kansas Ent. Soc. Jour. 13:51 and 54, 1940. New name for *Deltocephalus coquilletti* Van Duzee, 1890, a homonym of *Thamnotettix coquilletti* Van Duzee, 1890, by its being referred to *Thamnotettix* by Osborn and Ball, Iowa Acad. Sci. Proc. 4:221, 1897.

[California, coll. Coquillett.] "Described from three males and two females (No. 611)."

1 ♂, 1 ♀ — "California, Coquillett, Type," the female with determination label "*Deltocephalus Coquilletti* Van Duzee." The male and female with Beamer's "Neoholotype" and "Neoallotype" labels, respectively.

"Neoholotype male and neoallotype female, California, Coquillett, a pair of Van Duzee's cotypes in the Iowa State College Collection, Ames, Iowa," designated by Beamer, Kansas Ent. Soc. Jour. 13:54, 1940.

Lectotype male, the above indicated specimen, here designated.

*Colladonus atropunctatus* (Van D.)

(Fig. 4)

*Thamnotettix atropunctata* Van Duzee, Ent. Amer. 6:91, 1890.

[California, coll. Coquillett.] "Described from a single female (No. 630)."

1 ♀ — "California, Coquillett, 630, Type."

Holotype female, the above indicated specimen.

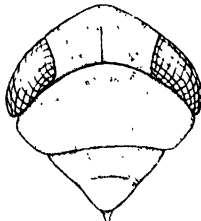


FIG. 4. *Colladonus atropunctatus* (Van Duzee). Head and thorax, dorsal view.

*Colladonus aureolus* (Van D.)

*Thamnotettix aureola* Van Duzee, Buffalo Soc. Nat. Sci. Bul. 5:213, 1894.

"California. Described from a single male example received from Mr. D. W. Coquillett labeled *Thamnotettix aureola*, Uhl."

1 ♂ — "California, Coquillett, 336, Type."

Holotype male, the above indicated specimen.

*Colladonus eburatus* (Van D.)

*Thamnotettix eburata* Van Duzee, Canad. Ent. 21:10, 1889.

[Canada, Muskoka Lake District.] "A male was swept from grass near a rivulet at Bracebridge; also taken in the vicinity of South Falls"

1 ♂, 1 ♀ — "Muskoka, Ont., July 1888, E. P. V. Coll., Type."

Lectotype male, the above indicated specimen, here designated.

*Colladonus flavocapitatus* (Van. D.)*Thamnotettix flavocapitata* Van Duzee, Ent. Amer. 6: 80, 1890.

[California, coll. Coquillett.] "Described from six males (No. 601) and three females (No. 154)."

2 ♂, 1 ♀—"California, Coquillett, Type."

Lectotype male, one of the above indicated specimens, here designated.

*Colladonus geminatus* (Van D.)*Thamnotettix geminata* Van Duzee, Ent. Amer. 6: 79, 1890.

[California, coll. Coquillett.] "Described from a single female example (No. 616)."

1 ♀—"California, Coquillett, 616, Type."

Holotype female, the above indicated specimen.

*Colladonus montanus* (Van D.)*Thamnotettix montanus* Van Duzee, Canad. Ent. 24: 268, 1892.

"British Columbia; Mountains of northwestern Colorado. Described from a fine pair received from Prof. Gillette and one male received from Mr. W. H. Harrington and labeled 'British Columbia.' This latter differs from the Colorado male in being more deeply coloured, with the transverse band between the eyes black, and showing two small transverse spots on the base of the front."

1 ♂, 1 ♀—"Col. Ac. Cat. 191, Type."

Lectotype male, the above indicated specimen, here designated.

*Commellus colon* (O. and B.)*Athysanus colon* Osborn and Ball, Iowa Acad. Sci. Proc. 4: 223, 1897.

[Iowa.] "Larvae were taken from *Stipa spartea* June 4th. and issued in the cages on the 6th. They were found up to June 10th, when they had all issued. Adults were taken through June and late into July . . ."

1 ♂, 1 ♀—"Ames, Ia. 6-23-96, Type."

1 ♂—"Ames, Ia. 7-1-96, Type."

1 ♂—"Ames, Ia. 7-2-96, Type."

1 ♂—"Ames, Ia. 7-14-96, Type."

1 ♀—"Ames, Ia. 6-4-96, Type."

1 ♀—"Ames, Ia. 6-13-96, Type."

1 ♀—"Ames, Ia. 6-20-96, Type."

Lectotype male, "Ames, Ia. 6-23-96, Type," here designated.

*Commellus comma* (Van D.)*Athysanus comma* Van Duzee, Canad. Ent. 24: 114, 1892.

"Iowa. One example received from Mr. C. P. Gillette."

1 ♀—"Ia., 2, Type," and with determination label "*Athysanus comma* Van Duzee."

Holotype female, the above indicated specimen.

*Commellus sexvittatus* (Van D.)

*Athysanus sexvittatus* Van Duzee, Canad. Ent. 26:93, 1894.

"Colorado. Described from two males received from Prof. C. P. Gillette."

1 ♂—"Colo. 633, Type," and with determination label "*Athysanus sexvittatus* Van D."

Lectotype male, the above indicated specimen, here designated.

*Davisonia punctifrons* var. *repleta* (Fieb.)

*Cicadula fasciifrons* var. *repleta* Fieber, Rev. d' Ent. 4:49, 1885.

*Cicadula punctifrons* var. *americana* Van Duzee, Canad. Ent. 23:169, 1891.

Concerning his *americana* Van Duzee states that "About Buffalo it occurs in great numbers on low willow bushes from June to August."

1 ♂, 3 ♀—"Lancaster, N. Y. 6-28-89, E. P. V. Coll., Type," mounted on a single pin, the male specimen uppermost.

8 ♀—"Lancaster, N. Y. 7-12-89, E. P. V. Coll., Type," five mounted on one pin and three on the other.

Lectotype male, the above indicated specimen, here designated for *americana* Van Duzee.

*Deltocephalus dentatus* (O. and B.)

*Athysanus dentatus* Osborn and Ball, Davenport Acad. Nat. Sci. Proc. 7:95, 1898.

"Described from eight females and one male from Colorado, one from Van Duzee collection, three from Prof. Pammel, and four females and one male from Prof. Gillette."

2 ♀—"Colo. 2220, Type."

1 ♀—"Colo. 429, Type."

1 ♀—"Colo. July '96, L. Pammel, Type."

Lectotype female, "Colo. 2220, Type," here designated.

*Deltocephalus flavicosta* (Stal)

*Jassus* (*Deltocephalus*) *flavicosta* Stal, Svensk. Vetensk. Handl. 2:53, 1862.

*Deltocephalus flavocostatus* Van Duzee, Canad. Ent. 24:116, 1892.

Van Duzee's *flavocostatus* was from "Mississippi. Described from two males received from Mr. Howard Evarts Weed."

1 ♂—"Miss., 30, Type," with determination label "*Deltocephalus flavocostatus* Van D."

1 ♂—"Miss., 31, Type."

Lectotype male, "Miss., 30, Type," here designated for *flavocostatus* Van Duzee.

*Deltocephalus flavocostatus* Van Duzee, 1892 = *Deltocephalus flavicosta* (Stal, 1862).

*Deltocephalus fuscinervosus* Van D.

Buffalo Soc. Nat. Sci. Bul. 5:207, 1894.

"California. Described from a single pair received from Mr. D. W. Coquillett labeled *Cicadula fuscinervosa*, Uhler, M. S."

1 ♂, 1 ♀—"California, Coquillett, Type," the female with determination label "*Deltocephalus fuscinervosus* Van D."

Lectotype male, the above indicated specimen, here designated.

*Deltocephalus minutus* Van D.

(Fig. 5)

Ent. Amer. 6:96, 1890.

[California, coll. Coquillett.] "Described from three males (No. 610; . . ."

1 specimen, abdomen missing but with ♂ sex symbol on pin—"California, Coquillett, Type," and with determination label "*Deltocephalus minutus* Van Duzee."

Lectotype male, the above indicated specimen, here designated.

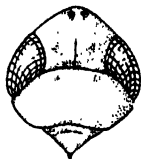


FIG. 5. *Deltocephalus minutus* Van Duzee. Head and thorax, dorsal view.

*Deltocephalus obesus* O. and B.

Davenport Acad. Nat. Sci. Proc. 7:81, 1898.

"Described from three macropterous and three brachypterous examples, two examples from Texas (Aaron), three from Orizaba, Vera Cruz (Osborn), and one from Arizona (Gillette)."

1 ♂—"Ariz. 2089, Gillette, Type."

2 ♀—"Orizaba, V. C., Mex. Jan. 9-16, '92, H. Osborn, Collector, Type."

2 ♀—"Texas, Aaron, Type."

Lectotype male, the above indicated specimen, here designated.

*Deltocephalus oculatus* Osborn and Ball, 1897 = *Laevicephalus unicoloratus* (G. and B., 1895).

*Deltocephalus punctatus* (O. and B.)

*Athysanus punctatus* Osborn and Ball, Davenport Acad. Nat. Sci. Proc. 7:94, 1898.

"Described from four females and two males swept from *Sporobolus*, Little Rock, Iowa, July 1st, and one female taken at Ames, Iowa, August 9th, and two females from Colorado through the kindness of Prof. Gillette."

1 ♂—"Exp. Sta., Ltl. Rck., Ia. 7-2-97, Type."

1 ♀—"Exp. Sta., Lttl. Rck., Ia. Jy. 2, '97, Type."

1 ♀—"Ltl. Rck., Ia. 7-2-97, H. Osborn, Collector, Type."

1 ♀—"Exp. Sta. 8-9-97, Ames, Ia., Type."

Lectotype male, the above specimen, here designated.

*Dorycephalus platyrhynchus* Osb.

Canad. Ent. 26:216, 1894.

"Described from two male specimens, one collected at Ames, Iowa, by Prof. C. P. Gillette, the other collected at West Point, Nebraska, by Prof. Laurence Bruner.

. . . . .

"Since forwarding the description of the male a special student in entomology, Mr. E. D. Ball, has brought in another male and the female here described."

No syntypes found in Iowa State College collection.

*Dorycephalus vanduzeei* O. and B.

*Dorycephalus vanduzeei* Osborn and Ball, Davenport Acad. Nat. Sci. Proc. 7:74, 1898.

"Described from two females collected at Little Rock, Iowa, July 1, 1897."

1 ♀ — "Exp. Sta., Ltlt. Rck., Ia. Jy. 2, '97."

The above indicated specimen and a female in the E. D. Ball material, United States National Museum, with identical pin label data but with a "Type" label attached, appear to be syntypes, even though the date is indicated as July 2 instead of July 1.

Lectotype female, the above indicated specimen in the Iowa State College collection, here designated.

*Draeculacephala manitobiana* Ball

Iowa Acad. Sci. Proc. 8:70, 1901.

"Described from eleven examples from Happy Hollow, North Park and Gunnison, Col., and a pair from Winnepeg, Manitoba."

1 ♀ — "North Pk., Col. 8-20-99, Type," with determination label "*Draeculacephala manitobiana* Ball, E D B."

The above indicated specimen is a syntype.

Lectotype female, "642, Winnipeg, Man.," from the E. D. Ball material in the United States National Museum, here designated.

*Driotura gammaroides* (Van D.)

*Athysanus gammaroides* Van Duzee, Buffalo Soc. Nat. Sci. Bul. 5:209, 1894.

"Described from a single female example captured in Madison Co., Kansas, by my brother M. C. Van Duzee. Another female from Colorado was in a lot received from Prof. C. P. Gillette."

1 ♀ — "Madison, K., M. C. V. Coll., Type," and with determination label "*Athysanus gammaroides* Van D."

Holotype female, the above indicated specimen.



*Driotura robusta* O. and B.

Davenport Acad. Nat. Sci. Proc. 7:87, 1898.

"Described from eight examples from Sioux City and Little Rock, Iowa, collected by the authors, and four examples from Colorado, received from Prof. Gillette."

1 ♂, 2 ♀ ♀ — "H. Osborn, Sx. Cty., Ia. July 7, '97, Type," one female with determination label "*Driotura robusta* O. & B."

1 ♀ — "Ltl. Rck., Ia. Jy. 2, '97, H. Osborn, Collector, Type," without "cotype" label.

Lectotype male, the above indicated specimen, here designated.

*Elymana inornata* (Van D.)

*Thamnotettix inornata* Van Duzee, Amer. Ent. Soc. Trans. 19:303, 1892.

"New York. Described from eight female examples captured at Lancaster during July and August."

1 ♀ — "Lancaster, N. Y. Aug. 1886, E. P. V. Coll., Type."

1 ♀ — "Lancaster, N. Y. 8-16-92, E. P. V. Coll., Type."

1 ♀ — "Lancaster, N. Y. 7-87, E. P. V. Coll., Type."

2 ♀ ♀ — "Lancaster, N. Y. Aug. 1887, E. P. V. Coll., Type."

1 ♀ — "Lancaster, N. Y. 7-23-89, E. P. V. Coll., Type."

Lectotype female, "Lancaster, N. Y. Aug. 1886, E. P. V. Coll., Type," here designated.

A male specimen of *Graminella fitchii* (Van D.), labeled as a "Cotype" of *inornata*, is believed to be a syntype of *fitchii*.

*Euscelis extrusus* (Van D.)

*Athysanus extrusus* Van Duzee, Canad. Ent. 25:283, 1893.

"New York; Connecticut. Described from three males. One taken at Portage Falls, N. Y., May 30th, 1888. The other two specimens were taken by me in Connecticut in the spring of 1883. One of these was swept from weeds and bushes on the hills about Northford, June 26th; the other, a very deeply coloured example, I found in a grove on Prospect St. in New Haven, June 4th."

1 ♂ — "Portage, N. Y., May 30th, '88 E. P. V. Coll., Type," with determination label "*Athysanus extrusus* Van D."

1 ♂ — "31, Type."

Lectotype male, "Portage, N. Y. May 30th, '88, E. P. V. Coll., Type," here designated.

*Eutettix luridus* (Van D.)

*Thamnotettix lurida* Van Duzee, Canad. Ent. 22:250, 1890.

"Described from two examples, a ♂ [sic] received from Prof. Osborn, labelled 'Ames, Iowa, May 19th, 1881,' and a ♀ [sic] from Mr. G. C. Davis labeled 'Agricultural College, Mich., Oct. 24th, 1888.'"

1 ♂ — "Ag. Coll., Mich., 10-24-88, 23, Type," with determination label "*Eutettix luridus* Van Duzee," and red label "Lectotype, *Eutettix luridus*"

(Van Duzee)." Genital structures dissected and contained in small glass vial attached to pin.

1 ♀—"In Woods, 5-19-81 Osborn, Ames, Ia., Type," with red label "Allotype, *Eutettix luridus* (Van Duzee)."

Lectotype male, the above indicated specimen, designated by Hepner (Univ. Kansas Sci. Bul. 28:288, 1942).

*Eutettix marmoratus* (Van D.)

Amer. Ent. Soc. Trans. 19:302, 1892.

"North Carolina. Collected on Mt. Balsam, July 23, 1890, by Mr. W. J. Palmer, Jr., . . ."

1 ♀—"Balsam, N. C., 7-23-90, W. J. P. Coll., Type," with determination label "*Eutettix marmoratus* Van D.," and red label "Lectotype, *Eutettix marmoratus* (Van Duzee)."

Lectotype female, the above indicated specimen, here designated. It is not clear from Van Duzee's original description how many specimens were at hand. Hepner (Univ. Kansas Sci. Bul. 28:290, 1942) is probably correct in considering the above specimen a holotype.

*Eutettix pictus* Van D.

Amer. Ent. Soc. Trans. 19:301, 1892.

"Pennsylvania. Described from a single female example which I owe to the kindness of Mr. C. W. Johnson, of Philadelphia."

1 ♀—"Pa., Type," with red label "Holotype, *Eutettix pictus* Van D.," determination label "*Eutettix pictus* Van D.," and red label "Lectotype, *Eutettix pictus* Van Duzee."

Holotype female, the above indicated specimen.

*Eutettix slossonae* Van D.

*Eutettix slossoni* Van Duzee, Buffalo Soc. Nat. Sci. Bul. 5:210, 1894.

"Described from one female specimen captured at Charlotte Harbor, Florida, by Mrs. Annie Trumbull Slosson to whom I take pleasure in dedicating this interesting form."

1 ♀—"Ch. Hbr., Fla., Type," with red label "Holotype, *Eutettix slossoni* Van D.," and determination label "*Eutettix Slossoni* Van D."

Holotype female, the above indicated specimen.

*Eutettix southwicki* Van D.

Buffalo Soc. Nat. Sci. Bul. 5:209, 1894.

"New York. Described from two male examples taken near New York City by Dr. E. B. Southwick . . ."

1 ♂—"E. B. Southwick [on pink paper with undecipherable notation], Type," with determination label "*Eutettix southwicki* Van D." red label "Lectotype, *Eutettix southwicki* Van Duzee." Genital structures dissected and contained in small vial attached to pin.

Lectotype male, the above indicated specimen, designated by Hepner (Univ. Kansas Sci. Bul. 28:289, 1942).

*Eutettix subaeneus* (Van D.)

*Thamnotettix subaenea* Van Duzee, Ent. Amer. 6:77, 1890.

[California, coll. Coquillett.] "Described from two females and one male (No. 223)."

1 ♂—"223, Type," with genital capsule removed, cleared, and contained in small vial attached to pin.

1 ♀—"California, Coquillett, Type," and with determination label "*Eutettix subaena* Van D."

Lectotype male, the above indicated specimen, designated by Hepner (Univ. Kansas Sci. Bul. 28:278, 1942).

*Flexamia abbreviata* (O. and B.)

*Deltocephalus abbreviatus* Osborn and Ball, Iowa Acad. Sci. Proc. 4:206, 1897.

[Iowa.] "Adults and full grown larvae were first taken in company with the preceding species [*pectinatus*] from *Bouteloa hirsuta* August 4th and 8th, 1896. By the middle of the month larvae had disappeared, adults continuing numerous throughout the month and on until the middle of September."

2 ♂ ♂, 3 ♀ ♀—"Ames, Ia. 8-15-96, Type."

1 ♂—"Ames, Ia. 8-4-96, Type."

1 ♂—"Ames, Ia. 8-11-96, Type."

1 ♀—"Ames, Ia. 9-17-96, Type."

Lectotype male, "Ames, Ia. 8-15-96, Type," here designated.

*Flexamia albida* (O. and B.)

*Deltocephalus albidus* Osborn and Ball, Iowa Acad. Sci. Proc. 4:201, 1897.

[Iowa.] "The larvae were first taken may 26th. They were then nearly fullgrown and remained abundant for two weeks, disappearing by the middle of June. The adults were taken the 3d of June, and by the middle were exceedingly abundant, continuing in decreasing numbers up to the middle of July. The only appearance of a second brood was the capture of an adult male August 18th."

2 ♂ ♂, 1 ♀—"Ames, Ia. 6-20-96, Type."

2 ♂ ♂, 1 ♀—"Ames, Ia. 6-3-96, Type."

1 ♀—"Ames, Ia. 6-15-96, Type."

1 ♀—"Ames, Ia. 7-21-96, Type."

Lectotype male, "Ames, Ia. 6-20-96, Type," here designated.

*Flexamia imputans* (O. and B.)

*Deltocephalus imputans* Osborn and Ball, Davenport Acad. Nat. Sci. Proc. 7:75, 1898.

"Described from thirty-two examples collected at Ames, Iowa."

1 ♂, 1 ♀—"Exp. Sta. Jy. 13, '97, Ames, Ia., Type."

2 ♂ ♂, 3 ♀ ♀—"Exp. Sta. Sept. 14, '97, Ames, Ia., Type."

Lectotype male, "Exp. Sta. Jy. 13, '97, Ames, Ia., Type," here designated.

*Flexamia inflata* (O. and B.)*Deltocephalus inflatus* Osborn and Ball, Iowa Acad. Sci. Proc. 4:202, 1897.

[Iowa.] "Adults have been taken rather sparingly through the last half of June, rather commonly through the first week in July, and one battered specimen the first of August."

1 ♂ — "Ames, Ia. 6-27-96, Type."

1 ♂ — "Ames, Ia. 6-15-96, Type."

1 ♂ — "Ames, Ia. 7-2-96, Type."

1 ♀ — "Ames, Ia. 7-8-96, Type."

1 ♀ — "Ames, Ia. 7-7-94, E. D. B., Type."

1 ♀ — "Ag. Coll., Ames, Ia., Type."

Lectotype male, "Ames, Ia. 6-27-96, Type," here designated.

*Flexamia pectinata* (O. and B.)*Deltocephalus pectinatus* Osborn and Ball, Iowa Acad. Sci. Proc. 4:205, 1897.

[Iowa.] "The first adults were taken May 26th, becoming more numerous up to the middle of June, then decreasing in numbers into July . . . The first larvae recognized as belonging to this species were taken August 4th. . . They were then nearly grown, and the adults were beginning to appear. Two weeks later the adults were abundant and the larvae gone. The adults continued abundant until into September, and could be found to the end of the season."

"This species was taken wherever *B. hirsuta* was found, and never anywhere else during the season."

1 ♂, 2 ♀ — "Ames, Ia. 6-4-96, Type."

1 ♂ — "Ames, Ia. 5-26-96, Type."

1 ♂ — "Ames, Ia. 8-11-96, Type."

1 ♂ — "Ames, Ia. 9-3-96, Type."

1 ♀ — "Ames, Ia. 8-18-96, Type."

Lectotype male, "Ames, Ia. 6-4-96, Type," here designated.

*Flexamia reflexa* (O. and B.)*Deltocephalus reflexus* Osborn and Ball, Iowa Acad. Sci. Proc. 4:203, 1897.

"It has been collected in abundance at Ames [Iowa] this season, and one Colorado example received from Professor Gillette."

1 ♂, 1 ♀ — "Ames, Ia. 6-23-96, Type."

1 ♂ — "Ames, Ia. 6-4-96, Type."

1 ♂ — "Ames, Ia. 6-13-96, Type."

1 ♂ — "Ames, Ia. 7-9-96, Type."

1 ♀ — "Ames, Ia. 6-15-96, Type."

1 ♀ — "Ames, Ia. 10-1-96, Type."

1 ♀ — "Ames, Ia. 10-12-96, Type."

Lectotype male, "Ames, Ia. 6-23-96, Type," here designated.

*Graminella fitchii* (Van D.)*Thamnotettix fitchii* Van Duzee, Ent. Amer. 6:133, 1890.

"Described from ten examples, representing both sexes, taken at Buffalo, Lancaster, and Colden, N. Y., and Welland County, Ontario,

from July 4th to September 10th, and one example from New Jersey (J. B. Smith)."

2 ♂ ♂, 2 ♀ ♀—"Lancaster, N. Y. 8-24-89, E. P. V. Coll., Type," mounted on one pin.

2 ♀ ♀—"Lancaster, N. Y. Aug. 1886, E. P. V. Coll."

1 ♂—"Colden, N. Y. July 4, 1886, E. P. V. Coll., Type" and bearing a red label "*Cotype, Thamnotettix inornatus*."

Lectotype male, "Lancaster, N. Y. 8-24-89, E. P. V. Coll., Type," the uppermost specimen on the pin, here designated.

The specimen from Colden, N. Y., is believed to be a syntype of *fitchii* rather than *inornatus* as it is labeled, since Colden is not listed as a locality for *inornatus*.

*Graminella nigrifrons* (Forbes)

*Cicadula nigrifrons* Forbes, 14th Rept. Illinois State Ent., p. 67, 1885.

*Thamnotettix perpunctata* Van Duzee, Buffalo Soc. Nat. Sci. Bul. 5:212, 1894.

Van Duzee's *perpunctata* was from "New York, N. Carolina, Mississippi. Described from numerous individuals of both sexes received from Mr. Howard Evarts Weed, taken in Miss. The N. C. Specimens were collected on Mt. Balsam, near Asheville, in July, by Mr. J. W. Palmer, Jr., of Buffalo, N. Y. Mr. E. B. Southwick has sent me examples from the vicinity of New York City and I have taken it about Buffalo in August."

2 ♂ ♂, 1 ♀—"Miss., Type."

3 ♀ ♀—"Miss."

1 ♂—"E. B. Southwick [on green paper], Type."

2 ♂ ♂, 5 ♀ ♀—"Balsam, N. C. 7-15-90, W. J. P. Coll., Type."

2 ♀ ♀—"Lancaster, N. Y. 8-24-89, E. P. V. Coll., Type."

Lectotype male, "Miss., Type," here designated for *perpunctata* Van Duzee.

*Graminella pallidula* (Osb.)

*Thamnotettix pallidula* Osborn, Iowa Acad. Sci. Proc. 5:245, 1898.

"Described from eight females and four males collected at Ames, Iowa, by Mr. E. D. Ball."

1 ♂, 1 ♀—"Exp. Sta., 8-20-97, Ames, Ia., Type."

1 ♂, 1 ♀—"Exp. Sta., Aug. 27, '97, Ames, Ia., Type."

4 ♀ ♀—"Exp. Sta., Sept. 14, '97, Ames, Ia., Type."

Lectotype male, "Exp. Sta. 8-20-97, Ames, Ia., Type," here designated.

*Graminella texana* (O. and B.)

*Athysanus texanus* Osborn and Ball, Davenport Acad. Nat. Sci. Proc. 7:92, 1898.

"Described from six examples from Texas (Aaron)."

4 ♀ ♀—"Texas, Aaron, Type."

Lectotype female, one of the above indicated specimens, here designated.

*Hebecephalus cruciatus* (O. and B.)

*Deltocephalus cruciatus* Osborn and Ball, Davenport Acad. Nat. Sci. Proc. 7:77, 1898.

"Described from thirty-two specimens, Little Rock, and Ames, Iowa."

1 ♂ — "Ames, Ia. 9-15-96, Exp. Sta., Type," with a red label "Hololectotype, *Hebecephalus cruciatus* (Osborn and Ball), Beamer and Tuthill." Genital structures dissected and mounted on celluloid tab attached to pin.

1 ♀ — "Ames, Ia. 9-7-96, Exp. Sta., Type."

2 ♀ ♀ — "Exp. Sta., Ltl. Rck., Ia. 7-2-97, Type."

1 ♀ — "Exp. Sta., Ltl. Rck., Ia., Jy. 2, '97, Type."

1 ♀ — "Ltl. Rck., Ia., Jy. 2, '97, H. Osborn, Collector, Type."

Lectotype male, the above indicated specimen, designated by Beamer and Tuthill (Univ. Kansas Sci. Bul. 22: 539, 1935).

*Hebecephalus signatifrons* (Van D.)

*Deltocephalus signatifrons* Van Duzee, Amer. Ent. Soc. Trans. 19: 305, 1892.

"Colorado. Described from one male and two female examples received from Mr. C. P. Gillette, and captured by him among the mountains in the northwestern part of the State."

1 ♂ — "Col. 158, 30, Type," with red label "Hololectotype, *Hebecephalus signatifrons* (Van Duzee), Beamer and Tuthill," and determination label "*Deltocephalus signatifrons* Van D." Genital structures dissected and mounted on a celluloid tab attached to pin.

1 ♀ — "Col., Ac. Cat. 68, Type," with red label "Allolectotype, *Hebecephalus signatifrons* (Van Duzee), Beamer and Tuthill."

Lectotype male, the above indicated specimen, designated by Beamer and Tuthill (Univ. Kansas Sci. Bul. 22: 533, 1935).

*Homalodisca liturata* Ball

Iowa Acad. Sci. Proc. 8: 48, 1901.

"Specimens are at hand from Phoenix, Ariz., Yuma, California, and Comondú, Lower Calif., Mexico."

1 ♀ — "Yuma, Cal., Wickham., Type," with determination label "*Homalodisca liturata* Ball, E D B."

The above specimen is a syntype.

Lectotype female, "Phoenix, Ariz. 5-97, Type," from the E. D. Ball material in the United States National Museum, here designated.

*Idiocerus amoenus* Van D.

*Idiocerus amoemus* Van Duzee, Canad. Ent. 26: 89, 1894.

*Idiocerus amoenus* Van Duzee, Calif. Univ. Pubs., Ent. 2: 575, 1917.

"Described from two female examples. One taken near Los Angeles, Cal., by Mr. D. W. Coquillett. The other, a more deeply coloured specimen, was sent me by Mr. C. P. Gillette, as an inhabitant of the mountains of northern Colorado, Mr. Coquillett's specimen came labeled *Idiocerus amoemus*, Uhler, a M. S. name which is quite appropriate to this pretty insect."

1 ♀—"California, Coquillett, 220, Type." Specimen parasitized by dryinid.

Lectotype female, the above indicated specimen, here designated.

*Idiocerus brunneus* O. and B.

(Figs. 6, A, B)

Davenport Acad. Nat. Sci. Proc. 7: 72, 1898.

"Described from numerous examples.

. . . . .

"It occurs abundantly on willows at Ames [Iowa], and has been received from Nebraska. There are two broods in a season, one in July and

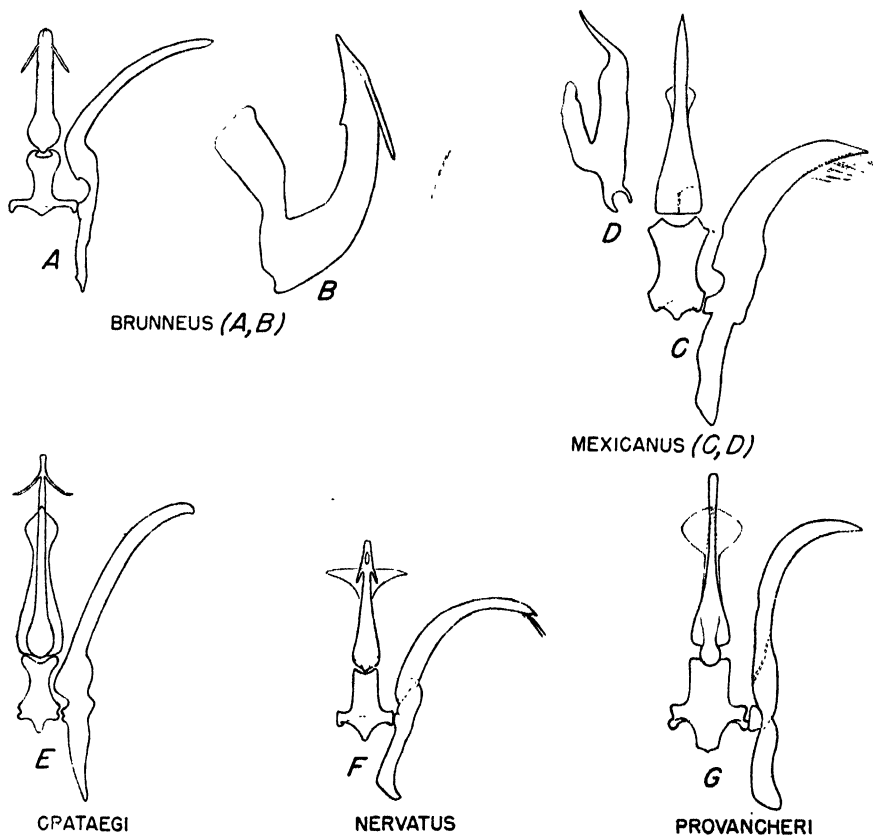


FIG. 6. *Idiocerus*. Internal male genitalia. A, style, connective, and aedeagus, ventral view; and B, aedeagus, lateral view, of *brunneus* O. and B. C, style, connective, and aedeagus, ventral view; and D, aedeagus, lateral view, of *mexicanus* O. and B. E, style, connective, and aedeagus, ventral view, of *crataegi* Van Duzee. F, style, connective, and aedeagus, ventral view, of *nervatus* Van Duzee. G, style, connective, and aedeagus, ventral view, of *provancheri* Van Duzee. Figure 6B shown at twice the magnification of remaining illustrations.

the other in September, the latter hibernating and depositing eggs in the spring, which hatch out by the first of June."

3 ♂ ♂, 2 ♀ ♀—"Ames, Ia. 9-22-96, Exp. Sta., Type."

1 ♂—"Sand Hills, Neb. July, Type."

1 ♀—"Ames, Ia. 9-30-96, Exp. Sta., Type."

1 ♀—"Exp. Sta. Sept. 18, '97, Ames, Ia., Type."

Lectotype male, "Ames, Ia. 9-22-96, Exp. Sta., Type," here designated. Genital capsule cleared and contained in small vial attached to pin.

*Idiocerus crataegi* Van D.

(Fig. 6, E)

Canad. Ent. 22:110, 1890.

"Buffalo, N. Y., July and August, occasionally on thorn bushes; Hamilton, Ont., James Johnston, Esq. Described from five male and six female examples."

3 ♂ ♂, 3 ♀ ♀—"Lancaster, N. Y. Aug. 1886, E. P. V. Coll., Type," mounted one male and two females on one pin, the remaining two males and one female on another pin.

1 ♀—"Buffalo, N. Y. 8-10-88, E. P. V. Coll., Type."

1 ♀—"Salamanca, N. Y. 5-2-89, Type," with labels bearing a manuscript name attached to pin.

Lectotype male, "Lancaster, N. Y. Aug. 1886, E. P. V. Coll., Type," uppermost specimen on pin bearing two males, here designated.

*Idiocerus mexicanus* O. and B.

(Figs. 6, C, D)

Davenport Acad. Nat. Sci. Proc. 7:133, 1898.

"Described from one female and one male collected by Prof. C. H. T. Townsend, Jicaltepec, Vera Cruz, Mexico, June 1896."

1 ♂—"San Rafael, Jicaltepec, Vera Cruz, June '96, Type ♂," with red label "Lectotype, *Idiocerus mexicanus* O. & Ball." Genital capsule cleared and contained in small vial attached to pin.

1 ♀—"San Rafael, Jicaltepec, Vera Cruz, June '96," with determination label "*Idiocerus mexicanus* O. & B., Type ♀," and red label "Allotype, *Idiocerus mexicanus* Osb. and Ball."

Lectotype male, the above indicated specimen, here designated.

*Idiocerus moniliferae* O. and B.

Davenport Acad. Nat. Sci. Proc. 7:71, 1898.

"Described from four females and one male. Larvae and adults taken from cottonwood in July." [At Ames, Iowa.]

1 ♀—"Exp. Sta., 6-19-97, Ames, Ia., Type."

1 ♀—"Exp. Sta., 7-29-97, Ames, Ia., Type."

1 ♀—"cttwd, Type."

Lectotype female, "Exp. Sta., 6-19-97, Ames, Ia., Type," here designated.



*Idiocerus nervatus* Van D.

(Fig. 6, F)

Buffalo Soc. Nat. Hist. Bul. 5:205, 1894.

"N. Y., N. J., Mich. Described from one male and four female examples: One pair taken by me at Lancaster, N. Y., June 28th, 1889; a female taken in New York City by Mr. E. B. Southwick, another taken at Anglesea, N. J., May 28th, by Prof. J. B. Smith and one from Agricultural College Mich., received from Mr. G. C. Davis."

1 ♂—"Lancaster, N. Y., E. P. V. Coll., Type." Genital capsule cleared and contained in small vial attached to pin.

1 ♀—"Lancaster, N. Y. 6-28-89, E. P. V. Coll., Type."

1 ♀—"Anglesea, N. J. 5-28, Type."

1 ♀—"E. B. Southwick, Type."

Lectotype male, the above indicated specimen, here designated.

*Idiocerus provancheri* Van D.

(Fig. 6, G)

*Idiocerus Provancheri* Van Duzee, Canad. Ent. 22:111, 1890.

"It is not uncommon here at Buffalo on oak and other bushes through June, July and August. I have also taken it at Muskoka, Ont., and have seen examples captured at Hamilton, Ont., by Mr. James Johnston."

1 ♂—"Buffalo, N. Y. June 1887, E. P. V. Coll." Genital capsule cleared and contained in small vial attached to pin.

1 ♂—"Lancaster, N. Y. 6-12-89, E. P. V. Coll."

1 ♀—"Muskoka, Ont. July 1888, E. P. V. Coll.," with note "no types of this species."

2 ♀ ♀—"Buffalo, N. Y. 8-10-88, E. P. V. Coll.," and with determination label "*Idiocerus Provancheri* Van Duzee."

Lectotype male, "Buffalo, N. Y. June 1887, E. P. V. Coll.," here designated.

According to my interpretation Van Duzee was naming a new species when he proposed the name *provancheri*, and the name is based upon specimens before him and not upon Provancher's specimens which that author had misidentified as *Jassus clitellarius* Say, 1831.

*Idiodonus belli* var. *gillettei* (Van D.)*Thamnotettix Gillettei* Van Duzee, Canad. Ent. 24:267, 1892.

"Colorado. Described from a single female example received from Prof. C. P. Gillette, . . ."

1 ♀—"Col., Ac. Cat 173, 162, Type."

Holotype female, the above indicated specimen.

*Idiodonus coquilletti* (Van D.)*Thamnotettix coquilletti* Van Duzee, Ent. Amer. 6:77, 1890.

[California, coll. Coquillett.] "Described from one male (No. 626) and two female (No. 331) examples."

1 ♀ — "California, Coquillett, Type."

Lectotype female, the above indicated specimen, here designated.

*Iowanus majestus* (O. and B.)

*Phlepsius majestus* Osborn and Ball, Iowa Acad. Sci. Proc. 4: 229, 1897.

"Specimens have been collected at Ames, and one specimen received from Philadelphia and another from Mississippi."

1 ♂, 1 ♀ — "Ames, Ia. 7-30-96, Type."

1 ♀ — "Ames, Ia. 8-18-93, E D B, Type." Abdomen missing.

1 ♀ — "Miss., Type," with determination label "*Phlepsius majestus* O. and B., types."

Lectotype male, "Ames, Ia. 7-30-96, Type," here designated.

*Iowanus spatulatus* (Van D.)

*Phlepsius spatulatus* Van Duzee, Amer. Ent. Soc. Trans. 19: 78, 1892.

"Described from five examples. One of these, wanting the abdomen, seems to be a male, the others are females. "Texas, Aaron," three examples; and "Ames, Iowa," one example, received from Mr. Osborn. One example without locality is in the lot set by Mr. Uhler. Two of Mr. Osborn's specimens are larger and fulvous-brown in color, . . ."

1 ♀ — "Texas, Aaron, Type."

1 ♀ — "Texas, Aaron" with determination label "*Phlepsius spatulatus* Van Duzee, Type." Abdomen missing.

1 ♀ — "Osborn, Ames, Ia.," with determination label "*Phlepsius spatulatus* Van Duzee" is *majestus* (O. and B.).

Lectotype female, "Texas, Aaron, Type," here designated.

*Laevicephalus cinerosus* (Van D.)

*Deltocephalus cinerosus* Van Duzee, Amer. Ent. Soc. Trans. 19: 305, 1892.

"California. Described from one male and four female examples received from Mr. D. W. Coquillett (No. 267), under Mr. Uhler's MS. name here adopted."

1 ♂, 2 ♀ — "California, Coquillett, Type," one female with determination label "*Deltocephalus cinerosus* Van D.," and male with genital capsule dissected and mounted on a celluloid tab attached to pin.

Lectotype male, the above indicated specimen, designated by Oman (Wash. Acad. Sci. Jour. 27: 477, 1937).

*Laevicephalus minimus* (O. and B.)

*Deltocephalus minimus* Osborn and Ball, Iowa Acad. Sci. Proc. 4: 211, 1897.

[Iowa.] "This . . . species occurred abundantly on a patch of raw prairie . . ."

1 ♂, 1 ♀ — "Ames, Ia. 10-7-96, Type."

1 ♂ — "Ames, Ia. 6-4-96, Type."

1 ♂ — "Ames, Ia. 9-15-96, Exp. Sta."

1 ♂ — "Ames, Ia. 10-1-96, Type."

2 ♀ — "Ames, Ia. 7-14-96, Type."

1 ♀ — "Ames, Ia. 9-22-96, Type."

Lectotype male, "Ames, Ia. 10-7-96, Type," here designated.

*Laevicephalus sylvestris* (O. and B.)

*Deltocephalus sylvestris* Osborn and Ball, Iowa Acad. Sci. Proc. 4:213, 1897.

"This is a widely distributed species, having been received from Maryland and Kansas. Specimens are in the Van Duzee collection from Ontario, and it has been taken at Ames for a number of years."

2 ♂ ♂, 2 ♀ ♀ — "Ames, Ia. 9-25-96, Type."

1 ♂ — "Ames, Ia. 10-12-96, Type."

1 ♀ — "Ames, Ia. 7-4-96, Type."

1 ♀ — "Ames, Ia. 7-11-96, Type."

Lectotype male, "Ames, Ia. 9-25-96, Type," here designated.

*Laevicephalus unicoloratus* (G. and B.)

*Deltocephalus unicoloratus* Gillette and Baker, Colorado State Agr. Coll., Agr. Exp. Sta. Bul. 31:89, 1895.

*Deltocephalus oculatus* Osborn and Ball, Iowa Acad. Sci. Proc. 4:212, 1897.

Osborn and Ball state concerning *oculatus* that, "This species has been received from Colorado, and has been collected at Ames [Iowa] prior to this season. It was first taken this year as adults the last week in May, and from then on through July. Larvae were taken abundantly during the second and third weeks in July, disappearing by the end; adults were again found from the middle of July through August; larvae again appearing in August, maturing through September; adults from the first of September on through the season.

"It has been found everywhere on *Andropogon scoparius*, to which it seems strictly confined."

1 ♂, 3 ♀ ♀ — "Ames, Ia. 10-1-96, Type."

1 ♂ — "Ames, Ia. 7-14-96, Type."

1 ♂ — "Ames, Ia. 8-31-96, Type."

1 ♂ — "Ames, Ia. 9-7-96, Type."

1 ♀ — "Ames, Ia. 6-4-96, Type."

Lectotype male, "Ames, Ia. 10-1-96, Type," here designated for *oculatus* O. and B.

*Limotettix parallelus* (Van D.)

*Athysanus parallelus* Van Duzee, Canad. Ent. 23:169, 1891.

"Described from one male and seven female examples, all taken near South Falls, on the Muskoka River, Ont., about the first of August."

1 ♂, 4 ♀ ♀ — "Muskoka, Ont. July 1888, E. P. V. Coll., Type."

Lectotype male, the above indicated specimen, here designated.

*Lonatura catalina* O. and B.

Davenport Acad. Nat. Sci. Proc. 7:83, 1898.

"Described from numerous examples of all the forms from Burlington, Ames, Sioux City, and Little Rock, Iowa, and from Yankton, S. D."

5 ♂ ♂, 1 ♀—"Exp. Sta., 6-24-97, Ames, Ia., Type," the female with determination label "*Lonatura catalina* O. and B." and the males without "Cotype" labels.

1 ♂, 1 ♀—"Exp. Sta., 6-14-97, Ames, Ia., Type."

1 ♂—"Exp. Sta., 7-16-97, Ames, Ia., Type."

1 ♂—"Exp. Sta., 8-4-97, Ames, Ia., Type," without "Cotype" label.

3 ♀ ♀—"Exp. Sta., 6-27-97, Ames, Ia., Type," one without "Cotype" label.

1 ♀—"Exp. Sta., Jy. 31, '97, Ames, Ia., Type."

1 ♀—"Ames, Ia. 8-15-96, Exp. Sta., Type."

1 ♀—"H. Osborn, Yankton, S. D. Jl. 6, '97, Type."

1 ♀—"H. Osborn, Brlington, Ia. Sep. 5, '97, Type."

Lectotype male, a brachypterous specimen, "Exp. Sta. 6-24-97, Ames, Ia., Type," here designated.

*Lonatura megalopa* O. and B.

*Lonatura* ? *megalopa* Osborn and Ball, Davenport Acad. Nat. Sci. Proc. 7:85, 1898.

"Described from nine males and thirteen females collected from a high knoll at Little Rock, Iowa, July 2d."

1 ♂—"Exp. Sta., Ltl. Rck., Ia. Jy. 2, '97, Type," with yellow label "Plesiotype, genitalia, R. H. Beamer," and red label "Lectoholotype, *Lonatura megalopa* Osborn and Ball." Genital structures dissected and contained in a small vial attached to pin.

2 ♂ ♂, 2 ♀ ♀—"Exp. Sta., Ltl. Rck., Ia. Jy. 2, '97, Type," one female with red label "Lectoallotype, *Lonatura megalopa* Osborn and Ball."

1 ♂, 2 ♀ ♀—"Exp. Sta., Ltl. Rck., Ia. 7-2-97, Type," one female with determination label "*Lonatura megalopa* O and B."

1 ♂—"Ltl. Rck., Ia. Jy. 2, '97, H. Osborn, Collector, Type."

1 ♀—"Exp. Sta. Jy. 13, '97, Ames, Ia., Type."

Lectotype male, the above indicated specimen, designated by Beamer (Kansas Ent. Soc. Jour. 11:31, 1938).

*Macropsis basalis* (Van D.)

*Pediopsis basalis* Van Duzee, Ent. Amer. 5:171, 1889.

"Described from a single female specimen, taken near Muskoka Lake [Canada], about the first of August, 1888."

1 ♀—"Muskoka Ont. July 1888, E. P. V. Coll., Type," with determination label "*Pediopsis basalis* Van D."

Holotype female, the above indicated specimen.

*Macropsis bifasciata* (Van D.)

*Pediopsis bifasciata* Van Duzee, Ent. Amer. 5:173, 1889.

"Described from one ♀ taken at Muskoka Lake the last of July 1888."

1 ♀ — "Muskoka, Ont. July 1888, E. P. V. Coll., Type," with determination label "*Pediopsis bifasciata* Van Duzee."

Holotype female, the above indicated specimen.

*Macropsis canadensis* (Van D.)

*Pediopsis flavescens* Van Duzee, nec Provancher, 1872, Ent. Amer. 5:173, 1889.

*Pediopsis canadensis* Van Duzee, Canad. Ent. 22:111, 1890. New name for *flavescens* Van Duzee, 1889, nec *Pediopsis flavescens* Provancher, 1872.

Concerning his *flavescens* Van Duzee states that, "Two examples; Muskoka, Ont., July 1888, and Lancaster, N. Y., June 27th 1889."

1 specimen, abdomen missing, presumably a female—"Muskoka Ont., July 1888, E. P. V. Coll., Type."

Lectotype [female?], the above indicated specimen, here designated.

*Macropsis ferruginoides* (Van D.)

*Pediopsis ferruginoides* Van Duzee, Ent. Amer. 5:171, 1889.

"Montana. Two examples, both females, received from Mr. Uhler. One, a pale individual, evidently immature, exhibits but traces of the yellow markings on the pronotum and scutellum."

1 ♀ — "Mon, Type," with determination label "*Pediopsis ferruginoides* V. D."

Lectotype female, the above indicated specimen, here designated.

*Macropsis fumipennis* (G. and B.)

*Pediopsis fumipennis* Gillette and Baker, Colorado State Agr. Coll. Agr. Exp. Sta. Bul. 31:73, 1895.

*Pediopsis crocea* Osborn and Ball, Davenport Acad. Nat. Sci. Proc. 7:68, 1898.

Osborn and Ball's *crocea* was "Described from eight females and five male collected on honey locust at Lexington, Ky., by H. Garman."

2 ♂ ♂, 8 ♀ ♀ — "Ky. Exp. St., H. Garman, Type."

Lectotype male, one of the above indicated specimens, here designated for *crocea* Osborn and Ball.

*Macropsis fumipennis* var. *gleditschiae* (O. and B.)

*Pediopsis gleditschiae* Osborn and Ball, Davenport Acad. Nat. Sci. Proc. 7:67, 1898.

[Iowa.] "Described from numerous examples.

. . . . .

"Found exclusively on the honey locust . . . , the larvae appearing in May and maturing before the middle of June, the adults throughout June and the first half of July."

2 ♂ ♂, 2 ♀ ♀ — "Exp. Sta., Jy. 7, '97, Ames, Ia., Type."

3 ♂ ♂, 1 ♀ — "Exp. Sta., 6-11-97, Ames, Ia., Type."

1 ♂, 1 ♀ — "Exp. Sta., 6-12-97, Ames, Ia., Type."

1 ♂, 4 ♀ ♀—"Exp. Sta., 6-22-97, Ames, Ia., Type."

1 ♀—"Exp. Sta., 7-7-97, Ames, Ia., Type."

Lectotype male, "Exp. Sta. Jy. 7, 97, Ames, Ia., Type," here designated for *gleditschiae* Osborn and Ball.

*Macropsis insignis* (Van D.)

*Pediopsis insignis* Van Duzee, Ent. Amer. 5:171, 1889.

"Many examples taken at Lancaster, N. Y., July 9th, 1889, on low bushes of Wild Plum. One ♀, taken at Madison, Kan., by my brother, M. C. Van Duzee, only differs from the eastern examples in the slightly darker color and abbreviated elytra which reach only to the top of the abdomen."

3 ♂ ♂, 5 ♀ ♀—"Lancaster, N. Y. 7-9-89, E. P. V. Coll., Type," mounted on four pins, as follows: 1 ♂, 1 ♀; 1 ♂, 2 ♀ ♀; 1 ♀; 1 ♂, 1 ♀. One pin bearing a male and female specimen has determination label "*Pediopsis insignis* Van D."

1 ♀—"Madison, K., M. C. V. Coll, Type."

Lectotype male, one of the above indicated specimens, here designated.

*Macropsis nubila* (Van D.)

*Pediopsis nubila* Van Duzee, Ent. Amer. 6:37, 1890.

[California, coll. Coquillett.] "Described from two female examples (N. 226)."

1 ♀—"California, Coquillett, 226, Type," with determination label "*Pediopsis nubila* Van D." and red label "*Pediopsis nubila* Van D., Holotype."

Lectotype female, the above indicated specimen, here designated.

*Macropsis occidentalis* (Van D.)

*Pediopsis occidentalis* Van Duzee, Psyche 5:238, 1889.

"California. Described from two females and three males. Nos. 602 ♂ and 603 ♀. Coquillett."

1 ♂—"California, Coquillett, 602, Type."

1 ♂—"California, Coquillett, Type."

1 ♂—"Calif., Type."

1 ♀—"California, Coquillett, Type," with determination label "*Pediopsis occidentalis* Van D."

Lectotype male, "California, Coquillett, 602, Type," here designated.

*Macropsis punctifrons* (Van D.)

*Pediopsis punctifrons* Van Duzee, Ent. Amer. 5:174, 1889.

"Arizona. Collected by the late H. K. Morrison. Described from seven examples; four received from Mr. Uhler and three from the Cornell University collection."

2 ♂ ♂, 1 ♀—"Arizona, C. U. Lot 34., Cornell U., Lot 45, Sub. 427, Type," both males on one pin and with determination label "*Pediopsis punctifrons* Van D."

1 ♀ — "Arizona, C. U. Lot 34., Type," with determination label "*Pediopsis punctifrons* Van D."

Lectotype male, the uppermost of the above indicated specimens, here designated.

*Macropsis reversalis* (O. and B.)

*Pediopsis reversalis* Osborn and Ball, Davenport Acad. Nat. Sci. Proc. 7: 69, 1898.

"Described from twenty-four males and twenty-four females collected from willows at Ames, Iowa, from the middle of June until into August, and three males from the Van Duzee collection from Colden, N. Y."

5 ♂ ♂, 3 ♀ ♀ — "Ames, Ia. 7-4-96, Exp. Sta., Type."

1 ♂, 2 ♀ ♀ — "Exp. Sta., Jy. 21, '97, Ames, Ia. Type."

1 ♀ — "Exp. Sta., Jy. 29, '97, Ames, Ia., Type."

1 ♀ — "Exp. Sta., 8-21-97, Ames, Ia., Type."

Lectotype male, "Ames, Ia., 7-4-96, Exp. Sta., Type," here designated.

*Macropsis sordida* (Van D.)

*Pediopsis sordida* Van Duzee, Canad. Ent. 26: 89, 1894.

"Colorado. Described from two male and five female examples collected among the Rocky Mountains by Prof. C. P. Gillette."

1 ♂ — "Colo., Ac. Cat. 203, Type."

1 ♀ — "Colo., 173, Type," with determination label *Pediopsis sordida* Van D."

1 ♀ — "Colo., Ac. Cat. 175, Type," without "cotype" label.

Lectotype male, the above indicated specimen, here designated.

*Macropsis suturalis* (O. and B.)

*Pediopsis suturalis* Osborn and Ball, Davenport Acad. Nat. Sci. Proc. 7: 67, 1898.

"Described from one male and seven females from Ames, Iowa, and one female from the Van Duzee Collection (Colden, N. Y.)."

1 ♂, 1 ♀ — "I A C, 6-12-94, C W M, Type." Head missing from male specimen.

1 ♀ — "Ames, Ia. 7-4-96, Exp. Sta., Type."

1 ♀ — "Exp. Sta. Jy. 29, '97, Ames, Ia., Type," with determination label "*Pediopsis suturalis* O. and B."

Lectotype female, "Ames, Ia. 7-4-96, Exp. Sta., Type," here designated.

*Macropsis tristis* (Van D.)

*Pediopsis tristis* Van Duzee, Canad. Ent. 22: 249, 1890.

"Described from three males collected by Prof. Herbert Osborn at Fairfax, Iowa, June 22nd and 24th, 1889, . . ."

1 ♂ — "IOWA, Fairfax, 6-24-89, Type," with determination label "*Pediopsis tristis* Van Duzee."

Lectotype male, the above indicated specimen, here designated.

*Macrosteles lepidus* (Van D.)

*Cicadula lepidus* Van Duzee, Canad. Ent. 26:139, 1894.

"Described from two female examples, Kansas, July, Prof. F. H. Snow. New York City, June, Mr. E. B. Southwick. Prof. Snow's specimen was taken at electric light, in Dodge Co., Kansas."

1 ♀—"July, taken at Electric Light, Dodge Co. Kas., U. of K. Col, Lot 65, Sub., 44, Type," with red label "Holotype, *Cicadula lepidus* Van D." attached to pin.

Lectotype female, the above indicated specimen, here designated.

*Macrosteles slossonae* (Van D.)

*Cicadula Slossoni* Van Duzee, Canad. Ent. 25:281, 1893.

"New York; New Hampshire. Described from three examples; one male taken by me at Lancaster, N. Y., July 12th, 1889, a female taken at "High Bridge," New York City, in June, by Mr. E. B. Southwick, and a second female taken on the summit of Mt. Washington by Mrs. Annie Trumbull Slosson, to whom I take pleasure in dedicating this pretty little species . . ."

1 ♂—"Lancaster, N. Y. 7-12-89, E. P. V. Coll., Type," and with red label "Lectotype, *Cicadula slossoni* V. D."

1 ♀—"E. B. Southwick [on pink paper], Type," and with faded purple paper tab on pin.

Lectotype male, the above indicated specimen, here designated.

*Menosoma cincta* (O. and B.)

*Eutettix cincta* Osborn and Ball, Davenport Acad. Nat. Sci. Proc. 7:97, 1898.

"Described from numerous examples of both sexes collected at Ames, Iowa, two specimens from Texas (Aaron), and one from Washington, D. C. (Heideman)."

1 ♂, 2 ♀—"Ames, Ia. 7-30-96, Exp. Sta., Type."

1 ♂—"Ames, Ia. 8-3-96, Exp. Sta., Type."

1 ♂—"Ames, Ia. 8-7-96, Exp. Sta., Type."

1 ♂—"Ames, Ia. 8-13-96, Exp. Sta., Type."

1 ♀—"Ames, Ia. 10-5-96, Exp. Sta., Type."

1 ♀—"Ames, Ia., Ac. Cat. 195, Type."

1 ♀—"Texas, Aaron, Type," without "cotype" label.

Lectotype male, "Ames, Ia. 7-30-96, Exp. Sta., Type," here designated.

*Mesamia straminea* (Osb.)

*Paramesus stramineus* Osborn, Iowa Acad. Sci. Proc. 5:241, 1898.

"Described from five females and one male. Of the females two were collected at Sioux City, July 7th, one at Sioux Falls, S. D., July 4th, and one at Ames, June 15th; and one collected at West Point, Neb., in June has been sent to me by Professor Bruner. The male was collected by Mr. Ball at Little Rock, Iowa, July 2d."



- 1 ♂—"Exp. Sta., Ltl. Rck., Ia. Jy. 2, '97, Type."  
 1 ♀—"Ames, Ia. 6-15-96, Exp. Sta., Type."  
 2 ♀—"H. Osborn, Sx. Cty., Ia. July 7, '97, Type."  
 1 ♀—"H. Osborn, Sux. Falls, S. D., Jl. 4, '97, Type."  
 1 ♀—"West Pt., Neb. 6-84, 85, Type."

Lectotype male, the above indicated specimen, here designated.

*Neocoelidia lactipennis* (Van D.)

(Figs. 7, A, B)

*Jassus lactipennis* Van Duzee, Ent. Amer. 6:49, 1890.

[California, coll. Coquillett.] "Described from one male (No. 629) and two female (No. 277) specimens."

1 ♂—"629, Type, *Jassus lactipennis* V. D." Genital capsule cleared and contained in small vial attached to pin.

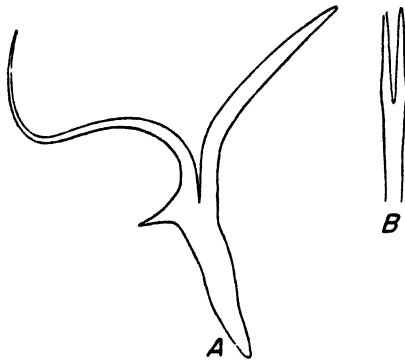


FIG. 7. *Neocoelidia lactipennis* (Van Duzee). Internal male genitalia. A, lateral view of aedeagus; B, dorso-caudal view of accessory process of aedeagus.

- 1 ♀—"California, Coquillett, Type."

Lectotype male, the above indicated specimen, here designated.

*Neokolla dolobrata* (Ball)

*Tettigonia hieroglyphica* var. *dolobrata* Ball, Iowa Acad. Sci. Proc. 8:52, 1901.

Concerning *dolobrata* Ball writes, "... *hieroglyphica*, and *dolobrata* ... are the only ones [forms] found in the Mississippi valley and as far west as central Kansas; they occur also in Texas, Arizona, and Mexico."

1 ♂—"Ames, Ia. 9-3-96, Exp. Sta.," with determination label "*Tettigonia hieroglyphica* var. *dolobrata* Ball, E D B," in Ball's handwriting.

2 ♂ ♂—"Ames, Ia. 9-22-96, Exp. Sta."

The above indicated specimens are considered syntypes.

Lectotype male, "E. D. Ball, 7-20-95, Ames, Ia., Type," from the E. D. Ball material in the United States National Museum, here designated.

*Neokolla uhleri* (Ball)

*Tettigonia hieroglyphica* var. *uhleri* Ball, Iowa Acad. Sci. Proc. 8:52, 1901.

Ball states that "The *uhleri* is the common form in Wyoming, Colorado, Arizona, and New Mexico, and extends westward to the coast."

1 ♂, 2 ♀ —“California, Coquillett,” one of the females with determination label “*Tettigonia hieroglyphica* var. *uhleri* Ball, E D B,” in Ball’s handwriting.

2 ♂ ♂ —“Arizona, C. U. Lot 34., Cornell U. Lot 45, Sub. 414,” on one pin with determination label as indicated above.

1 ♀ —“Col.,” with determination label as indicated above.

The above indicated specimens are considered syntypes.

Lectotype female, “Colo. 2253, Type,” from the E. D. Ball material in the United States National Museum, here designated.

*Nionia palmeri* (Van D.)

*Goniagnathus palmeri* Van Duzee, Canad. Ent. 23: 171, 1891.

“Described from a single female example taken at Mt. Balsam, N. C., Aug. 1st, 1890, by my friend Mr. W. J. Palmer, jr., . . . .”

1 ♀ —“Balsam, N. C., 8-1-90, W. J. P. Coll., Type,” and with determination label “*Goniagnathus Palmeri* Van Duzee.”

Holotype female, the above indicated specimen.

*Norvellina clarivada* (Van D.)

*Eutettix clarivada* Van Duzee, Canad. Ent. 26: 138, 1894.

“Colorado. Described from two male and four female examples received from Prof. C. P. Gillette.”

1 ♂ —“Colo. 566, Type,” with determination label “*Eutettix clarivadus* Van D.”

Lectotype male, the above indicated specimen, here designated.

*Norvellina scabra* (O. and B.)

*Eutettix scaber* Osborn and Ball, Davenport Acad. Nat. Sci. Proc. 7: 96, 1898.

“Described from three females and one male, collected at Ames, Iowa; two of them swept from white oak.”

1 ♂ —“Exp. Sta. 8-13-97, Ames, Ia., Type,” with red label “Lectotype, *Norvellina scaber* (O. and B.), ’38, det. D. R. Lindsay,” and determination label “*Norvellina scaber* (O. and B.), det. Lindsay ’38.” Genital structures dissected and contained in a small vial attached to pin.

1 ♀ —“Ames, Ia., Type,” with red label “Allotype, *Norvellina scaber* (O. and B.), det. ’38, D. R. Lindsay.” Abdomen missing.

1 ♀ —“Exp. Sta. 8-4-97, Ames, Ia., Type,” with yellow label “Plesio-type, *Norvellina scaber* (O. and B.), D. R. Lindsay.”

1 ♀ —“Exp. Sta., Jy. 7, ’97, Ames, Ia., Type.”

Lectotype male, the above indicated specimen, designated by Lindsay (Univ. Kansas Sci. Bul. 26: 189, 1939).

*Oncopsis cognatus* (Van D.)

(Figs. 8, A, B)

*Bythoscopus cognatus* Van Duzee, Ent. Amer. 6: 226, 1890.

“Described from two males and five female examples taken at Muskoka, Ont., July 1888. A pale greenish white ♀ taken at Lancaster, N. Y., May 31, 1877; is probably immature.”

1 ♂, 3 ♀ —“Muskoka, Ont., July 1888, E. P. V. Coll., Type,” two females on one pin. Male with genital capsule cleared and contained in a small vial attached to pin.

Lectotype male, the above indicated specimen, here designated.

The abdomen of the lectotype contains a dipterous larva, presumably of the family Dorilaidae. It is possible that this parasite has caused some modification of the genital structures, but none is apparent.

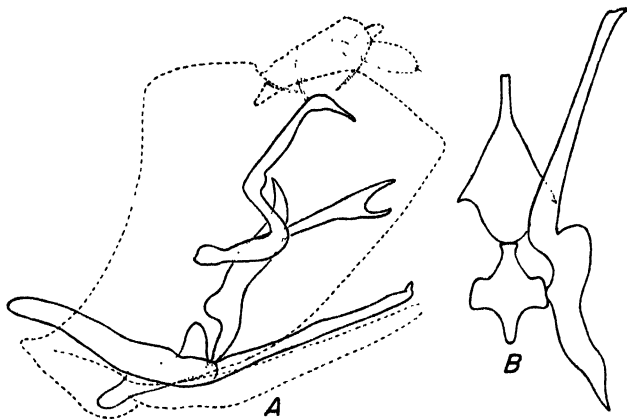


FIG. 8. *Oncopsis cognatus* (Van Duzee). Male genitalia. A, lateral view of genital capsule showing internal structures *in situ*; B, style, connective, and aedeagus, ventral view.

*Oncopsis verticis* (Say)

*Jassus verticis* Say, Philadelphia Acad. Nat. Sci. Jour. 6: 308, 1831.

*Bythoscopus distinctus* Van Duzee, Ent. Amer. 6: 224, 1890.

Van Duzee's *distinctus* was "Described from five male and nine female examples. Buffalo, one example swept from low bushes of *Populus grandidentata* July 10, 1889. Lancaster, N. Y., July and August. Niagara Falls, on oak M. C. Van Duzee. Maryland, June 11th, and Illinois, Uhler. Mt. Balsam, N. C., July, 1890, W. J. Palmer, Jr."

1 ♂ —“Niagra Falls, M. C. Van Duzee, Type.”

2 ♀ —“Lancaster N. Y. Aug. 1886, E. P. V. Coll., Type.”

1 ♀ —“Balsam N. C. 7-15-90, W. J. P. Coll., Type.”

1 ♀ —“Balsam N. C. 7-21-90, W. J. P. Coll., Type.”

Lectotype female, “Lancaster N. Y. Aug. 1886, E. P. V. Coll., Type,” here designated for *distinctus* Van Duzee.

*Osbornellus scalaris* (Van D.)

*Scaphoideus scalaris* Van Duzee, Ent. Amer. 6: 51, 1890.

[California, coll. Coquillett.] “Described from six individuals representing both sexes (No. 605 male, No. 623 female).”

1 ♂ —“California, Coquillett, Type, 605,” with red label “Lectoholotype, “*Osbornellus scalaris* E. P. Van Duzee.” Genital structures dissected and contained in a small vial attached to pin.

1 ♀ —“623, California, Coquillett, Type,” with red label “Lectoallotype, *Osbornellus scalaris* E. P. Van Duzee.”

Lectotype male, the above indicated specimen, designated by Beamer (Kansas Ent. Soc. Jour. 10:103, 1937).

*Paraphlepsius altus* (O. and B.)

*Phlepsius altus* Osborn and Ball, Iowa Acad. Sci. Proc. 4:228, 1897.

“This species has been collected at Ames and Little Rock, Iowa, and specimens are at hand from West Point, Neb. (Bruner).”

2 ♂ ♂, 2 ♀ ♀ —“Ames, Ia. 8-11-96, Type.”

2 ♂ ♂ —“Ames, Ia. 9-11-96, Type.”

1 ♀ —“Ames, Ia. 8-4-96, Type.”

Lectotype male, “Ames, Ia. 8-11-96, Type,” here designated.

*Paraphlepsius apertus* (Van D.)

*Phlepsius apertus* Van Duzee, Amer. Ent. Soc. Trans. 19:76, 1892.

“Described from one female and three male examples. One received from M. Provancher, taken near Quebec, the others taken by myself near Muskoka Lake, Ontario, in July, 1888.”

1 ♂, 1 ♀ —“Muskoka, Ont. July 1888, E. P. V. Coll., Type.”

1 ♂ —“Quebec, Provancher, Type.”

Lectotype male, “Muskoka, Ont. July 1888, E. P. V. Coll., Type,” here designated.

*Paraphlepsius cinereus* (Van D.)

*Phlepsius cinereus* Van Duzee, Amer. Ent. Soc. Trans. 19:68, 1892.

“Texas; Aaron. Described from one male and three female examples received from Prof. Herbert Osborn.”

1 ♂, 1 ♀ —“Texas, Aaron, Type,” the male with genital structures dissected and contained in a small vial attached to the pin.

Lectotype male, the above indicated specimen, here designated.

*Paraphlepsius fuscipennis* (Van D.)

*Phlepsius fuscipennis* Van Duzee, Amer. Ent. Soc. Trans. 19:70, 1892.

“Described from fourteen male and two female examples taken by Mr. E. B. Southwick near New York City in July, and one pair received from Mr. Uhler taken the first September.”

5 ♂ ♂ —“E. B. Southwick [on green paper], Type,” three of the pins with gray paper tabs. One specimen with genital structures dissected and contained in small vial attached to pin.

1 ♀ —“Sep 2, Type.”

Lectotype male, “E. B. Southwick, Type,” on a pin bearing a gray paper tab, here designated.

*Paraphlepsius humidus* (Van D.)

*Phlepsius humidus* Van Duzee; Amer. Ent. Soc. Trans. 19:76, 1892.

“Described from numerous examples of both sexes. . . . It is not uncommon about Buffalo from the last of July to the middle of September

in low swampy meadows and other humid situations. I have also taken it near Muskoka Lake, Ontario, . . . Mr. Uhler's material contains two or three examples labeled 'Delta R. R., September 15th.' Mr. E. B. Southwick also has taken it near New York City."

1 ♂, 2 ♀ ♀—"Lancaster N. Y. Aug. 1886, E. P. V. Coll., Type," the male with genital structures dissected and contained in a small vial attached to pin.

1 ♂—"Lancaster, N. Y. E. P. V. Coll., Type."

2 ♀ ♀—"Buffalo N. Y. Aug. 2d, 1886, E. P. V. Coll., Type."

1 ♀—"Ridgeway, Ont. Aug. 7th, 1886, E. P. V. Coll., Type."

Lectotype male, "Lancaster N. Y. Aug. 1886, E. P. V. Coll., Type," here designated.

*Paraphlepsius incisus* (Van D.)

*Phlepsius incisus* Van Duzee, Amer. Ent. Soc. Trans. 19: 73, 1892.

"Described from five male and two female examples; Buffalo and vicinity, July and August; Ridgeway, Ontario, Aug. 7, 1886."

2 ♂ ♂—"Buffalo, N. Y. 8-10-88, E. P. V. Coll., Type," mounted on one pin.

1 ♀—"Lancaster N. Y. Aug. 1886, E. P. V. Coll., Type."

Lectotype male, the uppermost of the above indicated specimens, here designated.

*Paraphlepsius latifrons* (Van D.)

*Phlepsius latifrons* Van Duzee, Amer. Ent. Soc. Trans. 19: 66, 1892.

"Described from a single pair taken at Odenton, Md., and received from Mr. Uhler. The male is labeled 'September 29th,' the female 'October 23d, pine.'"

1 ♀—"Odenton, Oct. 23, pine, Type," with red label "Allotype, *Phlepsius latifrons* Van D."

The above specimen is a syntype.

Lectotype male, "Odenton, Sept. 29," with name label, "*Phlepsius latifrons* V. Duz., Md.," in Uhler's handwriting, in the United States National Museum collection, here designated.

*Paraphlepsius lobatus* Osb.

*Phlepsius lobatus* Osborn, Iowa Acad. Sci. Proc. 5: 247, 1898.

"Described from one male and one female collected at Little Rock, Iowa, July 2, 1897, by Mr. E. D. Ball, and one female at Ames, Iowa, September 18th."

No syntypes found in Iowa State College collection.

*Paraphlepsius pallidus* (Van D.)

*Phlepsius pallidus* Van Duzee, Amer. Ent. Soc. Trans. 19: 69, 1892.

"Texas; Aaron. Described from a single female example received from Prof. Osborn."

One specimen, presumably a female, wings and portions of thorax only remaining—"Texas, Aaron, Type."

Holotype [female?], the above indicated specimen.

*Paraphlepsius punctiscriptus* (Van D.)

*Phlepsius punctiscriptus* Van Duzee, Amer. Ent. Soc. Trans. 19:75, 1892.

"Texas. Described from two female examples received from Mr. Uhler."

One specimen, abdomen missing—"Tex., Type."

Lectotype [female?], the above indicated specimen, here designated.

*Paraphlepsius solidaginis* (Walk.)

*Acocephalus solidaginis* Walker, List. Homop. Brit. Mus. 3:847, 1851.

*Phlepsius nebulosus* Van Duzee, Amer. Ent. Soc. Trans. 19:77, 1892.

Van Duzee's *nebulosus* was "Described from one male and two female examples. Of one pair received from Mr. Uhler the male is labeled 'Dacota, Rothauer,' and the female 'Mouse R.' The other female was received from Prof. Osborn, and is without a label."

1 ♀—"Type."

One specimen, abdomen missing—"Dacota, Rothauer, Type."

Lectotype female, the above indicated specimen labeled "Type," here designated for *nebulosus* Van Duzee.

*Paraphlepsius strobi* (Fitch)

*Bythoscopus strobi* Fitch, Ann. Rpt. State Cab. Nat. Hist. [N. Y.] 4:58, 1851.

*Phlepsius uhleri* Van Duzee, Amer. Ent. Soc. Trans. 19:67, 1892.

Van Duzee's *uhleri* was from "Odenton, Md., August 1st, Uhler. Described from a single male example."

Holotype male of *uhleri*, the above indicated specimen labeled, "Odent, Aug. 1, 7.," with name label, "*Phlepsius Uhleri* V. Duz., Md.," in Uhler's handwriting, and a red name label, "*Eutettix uhleri* (Van D.)," in C. F. Baker's handwriting, in United States National Museum collection.

*Paraphlepsius truncatus* (Van D.)

*Phlepsius truncatus* Van Duzee, Amer. Ent. Soc. Trans. 19:72, 1892.

"Described from one female and two male examples taken by Mr. W. J. Palmer, Jr., of this city, on Mt. Balsam, N. C., July 23, 1890."

1 ♀—"Balsam, N. C. 7-23-90, W. J. P. Coll., Type," with red label, "Holotype, *Phlepsius truncatus* Van. D."

1 ♂—"Balsam, N. C. 7-23-90, W. J. P. Coll., Type," with red label, "Allotype, *Phlepsius truncatus* Van D., ♂, " is *irroratus* (Say).

Lectotype female, the above indicated specimen, here designated.

*Pasadenus limbatus* (Van D.)

*Thamnotettix limbatus* Van Duzee, Ent. Amer. 6:92, 1890.

[California, coll. Coquillett.] "Described from a single male example (No. 612)."

1 ♂ — "California, Coquillett, 612, Type."

Holotype male, the above indicated specimen.

*Pediopsis crocea* O. and B., 1898 = *Macropsis fumipennis* (G. and B., 1895).

*Pediopsis flavescens* Van Duzee, 1889 = *Macropsis canadensis* (Van D., 1890).

*Pediopsis gleditschiae* Osborn and Ball, 1898 = *Macropsis fumipennis* var. *gleditschiae* (O. and B., 1898).

*Phlepsius nebulosus* Van Duzee, 1892 = *Paraphlepsius solidaginis* (Walk., 1851).

*Phlepsius uhleri* Van Duzee, 1892 = *Paraphlepsius strobi* (Fitch, 1851).

*Polyamia compacta* (O. and B.)

*Deltocephalus compactus* Osborn and Ball, Iowa Acad. Sci. Proc. 4: 217, 1897.

[Iowa.] "This species has been received from the state of Washington and collected at Ames the past season."

2 ♂ ♂ — "Ames, Ia. 6-27-96, Type."

1 ♂ — "Ames, Ia. 7-2-96, Type."

3 ♂ ♂ — "Ames, Ia. 7-9-96, Type."

Lectotype male, "Ames, Ia. 6-27-96, Type," here designated.

*Polyamia obtecta* (O. and B.)

*Deltocephalus obtectus* Osborn and Ball, Davenport Acad. Nat. Sci. Proc. 7: 78, 1898.

"Described from numerous specimens."

3 ♂ ♂, 2 ♀ ♀ — "Exp. Sta., 6-18-97, Ames, Ia., Type."

3 ♂ ♂ — "Exp. Sta., 6-12-97, Ames, Ia., Type."

Lectotype male, "Exp. Sta., 6-18-97, Ames, Ia., Type," here designated.

*Polyamia weedi* (Van D.)

*Deltocephalus Weedi* Van Duzee, Amer. Ent. Soc. Trans. 19: 306, 1892.

"Mississippi. Described from numerous examples received from Mr. Howard Evarts Weed, . . ."

2 ♂ ♂, 1 ♀ — "Miss., Type," one male with determination label, "*Deltocephalus Weedi* Van D."

Lectotype male, the above indicated specimen bearing the determination label.

*Prescottia lobata* (Van D.)

*Scaphoideus lobatus* Van Duzee, Buffalo Soc. Nat. Sci. Bul. 5: 211, 1894.

"New York. Described from a fine pair taken at Lancaster, N. Y. and a number of examples of both sexes taken by Mr. E. B. Southwick near New York City."

1 ♂, 1 ♀—"E. B. Southwick [on pink paper with undecipherable notation], Type."

1 ♀—"E. B. Southwick," on yellow paper.

1 specimen, wing only—"E. B. Southwick [on pink paper with undecipherable notation], Type."

Lectotype male, the above indicated specimen, here designated.

*Remadosus magnus* (O. and B.)

*Athysanus magnus* Osborn and Ball, Iowa Acad. Sci. Proc. 4: 225, 1897.

"It has been received from Texas, Kansas, Nebraska, Dakota, and northwest Iowa, also collected sparingly at Ames, from *Spartina cynosuroides* exclusively."

2 ♂, 1 ♀—"Ames, Ia. 8-12-96, Type."

1 ♂—"Ames, Ia. 8-8-96, Type."

1 ♂—"Dacota," with determination label in Ball's handwriting, "*Athysanus magnus* O. and B., Type."

1 ♀—"Ames, Ia. 8-19-95, Exp. Sta. C W M, Ac Cat. 2457, Type."

1 ♀—"Type."

Lectotype male, "Ames, Ia. 8-12-96, Type," here designated.

*Sanctanus sanctus* (Say)

*Jassus sanctus* Say, Philadelphia Acad. Nat. Sci. Jour. 6: 307, 1831.

*Scaphoideus picturatus* Osborn, Iowa Acad. Sci. Proc. 5: 243, 1898.

Osborn's *picturatus* was "Described from one female received from Prof. H. Garman, Lexington, Ky., and one male which I collected at Burlington [Iowa], September 5, 1897."

1 ♀—"Ky. Exp. St., H. Garman, 1802, Type," with red label, "*Scaphoideus picturatus* Osb., Holotype," and determination label, "*Scaphoideus picturatus* Osb., Type."

1 ♂—"H. Osborn, Brington, Ioa. 9-5-97, Type," with red label, "*Scaphoideus picturatus* Osb., Allotype," and determination label, "*Scaphoideus picturatus* Osb., Type."

Lectotype female, the above indicated specimen, here designated for *picturatus* Osborn.

*Scaphoideus luteolus* Van D.

Buffalo Soc. Nat. Sci. Bul. 5: 210, 1894.

"Described from one female taken at Anglesea, N. J., on July 16th by Prof. J. B. Smith, and three males captured near New York City, by Mr. E. B. Southwick, on July 6th, and August 12th, 1891."

1 ♀—"Anglesea, N. J. 7-16, Type," with red label, "Holotype, *Scaphoideus luteolus* Van D.," and determination label, "*Scaphoideus luteolus*, Van D."

1 ♂—"E. B. Southwick [on pink paper with undecipherable notation], Type," with red label, "Allotype, *Scaphoideus luteolus* Van D.," is probably a different species.

Lectotype female, the above indicated specimen, here designated.



*Scaphoideus ochraceus* Osb.

Iowa Acad. Sci. Proc. 5: 242, 1898.

"Described from twelve females and seven males collected at Ames from July 29th to August 13th."

1 ♂, 1 ♀—"Ames, Ia. 8-3-96, Exp. Sta., Type."

2 ♂ ♂, 1 ♀—"Ames, Ia. 7-29-96, Exp. Sta., Type."

2 ♀ ♀—"Ames, Ia. 8-13-96, Exp. Sta., Type," one specimen with determination label, "*Scaphoideus ochraceus* Osb., Type."

Lectotype male, "Ames, Ia. 8-3-96, Exp. Sta., Type," here designated.

*Scaphoideus productus* Osb.

Cincinnati Soc. Nat. Hist. Jour. 19: 200, 1900.

"Described from seven ♀s and two ♂s of which three females were taken at Ames, Iowa, August 3, 1896, August 7, 1897, and October 14, 1896. One at Sioux City, July 7, 1897. Three ♀s and one ♂ Onaga Kans. (Crevecoeur) and one ♂ from Kentucky (Garman)."

1 ♀—"Exp. Sta. Jy. 7, '97, Ames, Ia. Type," with determination label, "*Scaphoideus productus* Osb., H. O., Type."

The above specimen is a syntype, the lectotype having been designated in the Osborn collection at the Ohio State University by DeLong and Beery (Ohio Jour. Sci. 36: 341, 1936).

*Scaphytopius elegans* (Van D.)

*Platymetopius elegans* Van Duzee, Ent. Amer. 6: 94, 1890.

[California, coll. Coquillett.] "Described from a single female example (No. 610)."

1 ♀—"610, California, Coquillett, Type."

Holotype female, the above indicated specimen.

*Scleroracrus anthracinus* (Van D.)

*Athysanus anthracinus* Van Duzee, Canad. Ent. 26: 136, 1894.

"Iowa, Kansas and Colorado. Described from one female and two male examples. The Kansan specimen was captured at Madison, by M. C. Van Duzee. That from Iowa I owe to the kindness of Prof. Herbert Osborn, and the example from Colorado is from Prof. C. P. Gillette. Prof. Osborn's specimen came labeled "*Conogonus gagates*, Ashm., and in the National Museum is an example labeled *Scleroracrus anthracinus*, Uhler."\*

\* According to my interpretation, Van Duzee, by his association of *Conogonus gagates* and *Scleroracrus anthracinus* with the description of his *Athysanus anthracinus* thereby validated both the generic names *Conogonus* and *Scleroracrus* and the specific name *gagates*. The specific name *gagates* will remain as a synonym of *anthracinus*. *Conogonus* and *Scleroracrus* are available for use as monotypic generic names,

1 ♂—"Ia., Ac. Cat. 370, Gillette, Type," with determination labels "*Conogonus gagates* Ash." and "*Athysanus anthracinus* Van Duzee."

1 ♀—"Madison, K., M. C. V. Coll, Type," with determination label "*Scleroracrus anthracinus* Uhl."

Lectotype male, the above indicated specimen, here designated for *gagates* Van Duzee and *anthracinus* Van Duzee.

*Scleroracrus instabilis* (Van D.)

(Figs. 9, A, B, C)

*Athysanus instabilis* Van Duzee, Canad. Ent. 25: 284, 1893.

"Michigan; Colorado. Described from one male and three female examples taken at Agricultural College, Michigan, by my friend Mr. G. C. Davis, and another female received from Prof. C. P. Gillette, taken in Colorado."

1 ♂—"Ag. Coll., Mich. 8-23-'93, 184, Type." Genital capsule cleared and contained in a small vial attached to pin.

1 ♀—"Ag. Coll., Mich. 8-21-'93, 181, Type," and with determination label "*Athysanus instabilis* Van D."

Lectotype male, the above indicated specimen, here designated.

*S. instabilis* (Van D.) is usually considered a synonym of *cornicula*

and I have selected the latter for the generic concept commonly called *Ophiola*. The generic synonymy is as follows:

*Scleroracrus* Van Duzee, 1894, type, by monotypy, *Athysanus anthracinus* Van Duzee, 1894.

*Conogonus* Van Duzee, 1894, type, by monotypy, *Athysanus anthracinus* Van Duzee, 1894.

*Ophiola* Edwards, 1922, type, by original designation, *Cicada striatula* Fallén, 1826.

In addition to *anthracinus* and its synonym *gagates*, the following names, all representing new combinations, are referred to *Scleroracrus*:

*angustatus* (Osborn, 1915) [*Athysanus*]  
*arctostaphyli* (Ball, 1899) [*Athysanus*]  
*bullatus* (Ball, 1902) [*Thamnotettix*]  
*cacheolus* (Ball, 1938) [*Ophiola*]  
*calvatus* (Ball, 1916) [*Athysanus*]  
*comptonianus* (Ball, 1928) [*Ophiola*]  
*corniculus* (Marsh, 1866) [*Jassus*]  
*elongatus* (Osborn, 1915) [*Athysanus*]  
*finitimus* (Van Duzee, 1925) [*Euscelis*]  
*gentilis* (Van Duzee, 1925) [*Euscelis*]  
*glomerosus* (Ball, 1910) [*Thamnotettix*]  
*humidus* (Osborn, 1915) [*Athysanus*]  
*instabilis* (Van Duzee, 1893) [*Athysanus*]  
*luteolus* (Sleesman, 1929) [*Ophiola*]  
*osborni* (Ball, 1938) [*Ophiola*]  
*shastus* (Ball, 1916) [*Athysanus*]  
*speculatus* (Ball, 1928) [*Ophiola*]  
*striatulus* (Fallén, 1826) [*Cicada*]  
*symphoricarpae* (Ball, 1901) [*Athysanus*]  
*uhleri* (Ball, 1911) [*Athysanus*]  
*vaccinii* (Van Duzee, 1890) [*Athysanus*]  
*varus* (Ball, 1901) [*Athysanus*]

*Euscelis cuneatus* Sanders and DeLong, 1920, is referred to the genus *Limotettix* New combination.

(Marsh), but because of the very uncertain status of species in this genus it seems best at this time to treat *instabilis* as distinct.

*Scleroracrus vaccinii* (Van D.)

(Figs. 9, G, H, I)

*Athysanus vaccinii* Van Duzee, Ent. Amer. 6:135, 1890.

"New Jersey. Described from five male and four female examples kindly furnished me by Prof. J. B. Smith."

1 ♂, 1 ♀—"N. Jersey, J. B. Smith," the male with determination label "*Athysanus striatulus* ? Fall," in Van Duzee's handwriting. Male with genital capsule cleared and contained in a small vial attached to pin.

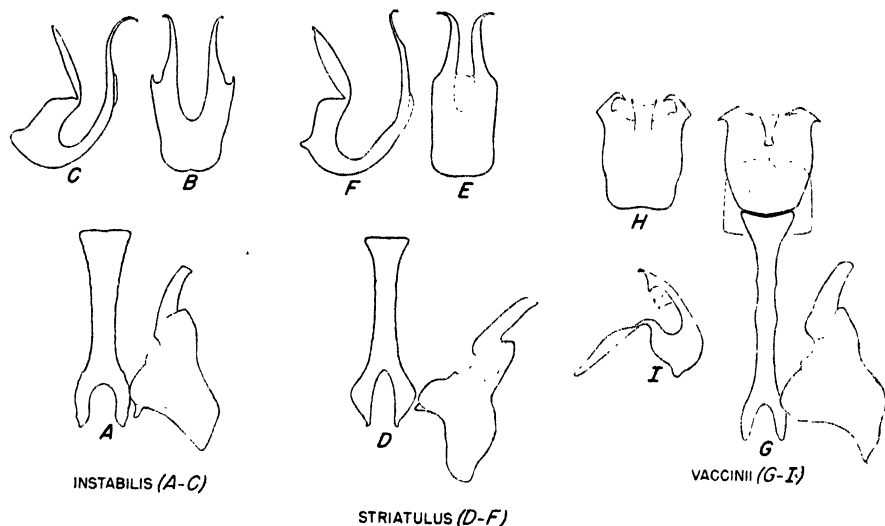


FIG. 9. *Scleroracrus*. Internal male genitalia. A, style and connective, ventral view; B, aedeagus, caudal view; and C, aedeagus, lateral view, of *instabilis* (Van Duzee). D, style and connective, ventral view; E, aedeagus, caudal view; and F, aedeagus, lateral view, of *striatulus* (Fallén). G, style, connective, and aedeagus, ventral view; H, aedeagus, caudo-ventral view; and I, aedeagus, lateral view, of *vaccinii* (Van Duzee).

Lectotype male, the above indicated specimen, here designated.

*Scleroracrus vaccinii* (Van Duzee) is usually considered to be a synonym of the European *striatulus* (Fallén), but the accompanying drawings illustrate the differences in the structure of the male genitalia of the two species. *S. vaccinii* belongs to a different species group than does *striatulus*. The illustrations of *striatulus* (fig. 9, D, E, F) were made from specimens from Sweden determined by Dr. Frej Ossiannilsson.

*Stirellus bicolor* (Van D.)

*Athysanus bicolor* Van Duzee, Canad. Ent. 24:114, 1892.

"Described from two female examples, one from Mississippi, kindly given me by Mr. Howard Evarts Weed, and a smaller specimen taken near Emporia, Kansas, by my brother Mr. M. C. Van Duzee."

1 ♀—"Miss., 24, Type," with determination label "*Athysanus bicolor* Van D."

Lectotype female, the above indicated specimen, here designated.

A second specimen, with pin label data "Miss., Ag. Coll." and bearing a "cotype" label, is not considered to be a syntype.

*Stirellus obtutus* (Van D.)

*Athysanus obtusus* Van Duzee, Canad. Ent. 24:115, 1892.

*Athysanus obtutus* Van Duzee, Canad. Ent. 24:156, 1892.

"Mississippi. Described from one male and three female examples received from Mr. Howard E. Weed."

2 ♀ ♀—"Miss., Type."

Lectotype female, one of the above indicated specimens, here designated.

A third pin, bearing the same data and a determination label, has the notation "Spm. missing" written across the name label.

*Stragania apicalis* (O. and B.)

*Macropsis apicalis* Osborn and Ball, Davenport Acad. Nat. Sci. Proc. 7:64, 1898.

"Described from numerous examples collected from the honey locust at Ames and Sioux City, Iowa, and one from West Point, Neb."

2 ♂ ♂, 4 ♀ ♀—"Exp. Sta., Jy. 7, '97, Ames, Ia., Type."

1 ♂, 2 ♀ ♀—"Exp. Sta., Jy. 1, '97, Ames, Ia., Type."

1 ♂, 1 ♀—"H. Osborn, Sx. Cty. Ia., July 7, '97, Type."

1 ♀—"Exp. Sta., 7-1-97, Ames, Ia., Type."

1 ♀—"Exp. Sta., Oct. 6, '97, Ames, Ia., Type."

Lectotype male, "Exp. Sta., Jy. 7, '97, Ames, Ia., Type," here designated.

*Texananus decorus* (O. and B.)

*Phlepsius decorus* Osborn and Ball, Iowa Acad. Sci. Proc. 4:230, 1897.

"Described from one male from Lincoln, Neb. (Bruner), and one female collected at Ames, Iowa."

1 ♂—"Lincoln, Neb. October 2-92, Type."

Lectotype male, the above indicated specimen, here designated.

*Texananus ovatus* (Van D.)

*Phlepsius ovatus* Van Duzee, Amer. Ent. Soc. Trans. 19:79, 1892.

"Texas. Described from two female examples received from Mr. Uhler."

1 specimen, forewings and abdomen missing—"Tex., Type."

Lectotype [female?], the above indicated specimen, here designated.

*Texananus superbus* (Van D.)

*Phlepsius superbus* Van Duzee, Amer. Ent. Soc. Trans. 19:81, 1892.

"North Carolina and Arizona. Described from one female and two male examples."

1 ♂ — "Arizona, U. Lot, 5," with determination label "*Phlepsius superbus* Van Duzee, type," and red label "Holotype, *Phlepsius superbus* Van D."

1 ♂ — "N. C., Type," without "Cotype" label.

1 specimen, abdomen and part of thorax missing — "Arizona, C. U. Lot 34, Cornell U. Lot 45, Sub. 435," with red label "Allotype, *Phlepsius superbus* Van D."

Lectotype male, "Arizona, U, Lot, 5," here designated.

*Thamnotettix Gillettii* Van Duzee, 1892 = *Idiodonus belli* var. *gillettei* (Van D., 1892).

*Thamnotettix perpunctata* Van Duzee, 1894 = *Graminella nigrifrons* (Forbes, 1885).

*Tinobregmus vittatus* Van D.

Buffalo Soc. Nat. Sci. Bul. 5: 214, 1894.

"Florida. Described from two female specimens received from Mr. C. W. Johnson of Philadelphia."

1 ♀ — "Fla., Type."

Lectotype female, the above indicated specimen, here designated.

*Tropicanus costomaculatus* (Van D.)

*Allygus costomaculatus* Van Duzee, Buffalo Soc. Nat. Sci. Bul. 5: 207, 1894.

"Described from two females received from Prof. Herbert Osborn and labeled "Texas Aaron."

2 ♀ ♀ — "Texas, Aaron, Type."

Lectotype female, one of the above indicated specimens, here designated.

*Ulopa canadensis* Van Duzee, 1892 = *Agallia quadripunctata* (Prov., 1872).

*Xestocephalus coronatus* O. and B.

Iowa Acad. Sci. Proc. 4: 184, 1897.

"Two males and one female . . . were taken from a deeply shaded patch of bluegrass in August. Ames, Iowa."

1 ♂ — "Ames, Ia. 8-17-96, Exp. Sta."

1 ♂, 1 ♀ — "Exp. Sta. 8-20-97, Ames, Ia."

Lectotype male, "Ames, Ia. 8-17-96, Exp. Sta." here designated.

None of the above specimens bears the characteristic "Type" label and may not be syntypes. However, the male designated above would appear to have been at hand at the time the description was drawn. A third male, "Exp. Sta., 9-2-97, Ames, Ia.," is definitely eliminated as a possible syntype.

*Xestocephalus pulicarius* Van D.

(Figs. 10, A, B)

Buffalo Soc. Nat. Sci. Bul. 5: 215, 1894.

"New York, Canada. This pretty little insect is sometimes abundant in August and Sept. about Buffalo in swampy pastures where *Carex vul-*

*pinoidea* grows. I have also taken it at Ridgeway, Ont., and Mr. E. B. Southwick has sent me two examples captured near New York City in August."

2 ♂ ♂—"Lancaster, N. Y. Aug. 1886, E. P. V. Coll., Type," mounted on one pin which bears a red label "Lectotype, *Xestocephalus pulicarius* Van D."

1 ♂—"Lancaster, N. Y. Aug. 1887, E. P. V. Coll., Type."

2 ♀ ♀—"Lancaster, N. Y. Aug. 1886, E. P. V. Coll., Type."

Lectotype male, "Lancaster, N. Y. Aug. 1886, E. P. V. Coll., Type," the uppermost specimen, here designated.

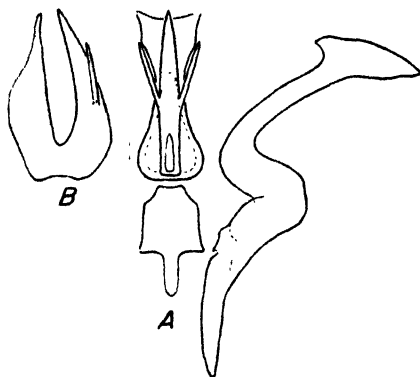


FIG. 10. *Xestocephalus pulicarius* Van Duzee. Internal male genitalia. A, style, connective, and aedeagus, ventral view; B, aedeagus, lateral view.

Additional specimens at hand, without either "Type" or "Cotype" labels, are probably syntypes. These are—

3 ♀ ♀—"Lancaster, N. Y. Aug. 1886, E. P. V. Coll.," mounted on one pin which bears determination label "*Xestocephalus pulicarius* Van D."

2 ♂ ♂—"Lancaster, N. Y. 9-3-88, E. P. V. Coll."

1 ♂—"Lancaster, N. Y. Aug. 1886, E. P. V. Coll."

1 ♂—"Lancaster, N. Y. 7-24-89, E. P. V. Coll."

1 ♂—"E. B. Southwick, N. B., 8-12-91."

1 ♀—"Ridgeway, Ont. Aug. 7th. 1886, E. P. V. Coll."

*Xestocephalus superbus* (Prov.)

*Deltoccephalus superbus* Provancher, Petite Faune Ent. Canad. 3: 339, 1890.

*Xestocephalus fulvocapitatus* Van Duzee, Buffalo Soc. Nat. Sci. Bul. 5: 215, 1894.

Van Duzee's *fulvocapitatus* was from "New York. Of this species I have taken one male and four female examples at Lancaster, N. Y., in August and September . . ."

1 ♂—"Lancaster, N. Y. Aug. 1886, E. P. V. Coll., Type," with red label "Lectotype, *Xestocephalus fulvocapitatus* Van D." attached.

2 ♀ ♀—"Lancaster, N. Y. 9-3-88, E. P. V. Coll., Type," with determination label "*Xestocephalus fulvocapitatus* Van D." attached.

Lectotype male, the above indicated specimen, here designated for *fulvocapitatus* Van Duzee.

*Xestocephalus tessellatus* Van D.

Buffalo Soc. Nat. Sci. Bul. 5:216, 1894.

"Charlotte Harbor, Florida, Mrs. Annie Trumbull Slosson; Mississippi, Howard Evarts Weed; Texas, "Aaron." Described from one male and four female specimens."

1 ♂ — "Miss., Type," and with red label "Holotype, *Xestocephalus tessellatus* V. D."

1 ♀ — "Miss., Type," and with determination label "*Xestocephalus tessellatus* V. D."

1 ♀ — "CH. HBR. FLA, Type."

1 ♀ — "Texas, Aaron."

Lectotype male, the above indicated specimen, here designated.

SUPERFAMILY FULGOROIDEA

FAMILY DELPHACIDAE

*Delphacodes campestris* (Van D.)

*Liburnia campestris* Van Duzee, Buffalo Soc. Nat. Sci. Bul. 5:254, 1897.

*Liburnia osborni* Van Duzee, Buffalo Soc. Nat. Sci. Bul. 5:250, 1897. New synonymy.

Van Duzee's *campestris* was from "New York, Ontario. Described from numerous examples of both sexes; Buffalo and vicinity, May to August, Portage Falls, May 31st; Ridgeway and Muskoka, Ont. about August 1st. Mississippi, H. E. Weed; New Hampshire, C. E. Weed; Michigan, Davis."

*Osborni* was from "Ocean Co., New Jersey in May, Prof. J. B. Smith; Agricultural College, Mich. G. C. Davis; Fairfax and Ames, Iowa, Prof. Herbert Osborn, . . . Also taken by me at Lancaster, N. Y. in August, 1880."

The following syntypes of *campestris* are at hand:

2 ♂ ♂ — "Buffalo, N. Y. 6-11-88, E. P. V. Coll., Type," mounted on one pin and with determination label "*Liburnia campestris* Van D." Brachypterous.

4 ♂ ♂, 2 ♀ ♀ — "Lancaster, N. Y. 8-2-90, E. P. V. Coll., Type," mounted on one pin. Brachypterous.

Lectotype male, "Buffalo, N. Y. 6-11-88, E. P. V. Coll., Type," lowermost specimen on pin, here designated for *campestris* Van Duzee.

The following syntypes of *osborni* are at hand:

1 ♂, 1 ♀ — "Ag. Coll., Mich., 205, Type," the male with determination label "*Liburnia Osborni* n sp." Macropterous.

1 ♂ — "Ocean Co., N. J. May, Type." Macropterous.

Lectotype male, "Ag. Coll., Mich., 205, Type," here designated for *osborni* Van Duzee.

Although the specific name *osborni* has page priority over *campestris* the latter name is more widely used and its identity better established, hence its choice as the name for this species.

The following specimens are probably syntypes of *osborni*:

2 ♀ ♀ — "Osborn, Ames, Ia."

1 ♂ — "Ag. Coll., Mich., 306."

1 ♀ — "Ocean Co., N. J. May."

1 ♂, 1 ♀ — "Fairfax, Ia. June 24, '89."

*Delphacodes consimilis* (Van D.)

*Liburnia consimilis* Van Duzee, Buffalo Soc. Nat. Sci. Bul. 5:249, 1897.

"California and Colorado. Described from three males from near Los Angeles, Calif. received from Mr. D. W. Coquillett under the name of *Delphax consimilis*, Uhler, M. S. and one pair taken in the mountains of north west Colorado by Prof. C. P. Gillette."

1 ♂ — "344, Type," with determination label "*Liburnia consimilis* Van D." Macropterous.

1 ♂ — "Type." Macropterous.

1 ♀ — "Colo. 633, Type." Macropterous.

Lectotype male, "344, Type," here designated.

*Delphacodes detecta* (Van D.)

*Liburnia detecta* Van Duzee, Buffalo Soc. Nat. Sci. Bul. 5:248, 1897.

"New York. Described from one pair received from Mr. E. B. Southwick, taken in New York City."

1 ♂, 1 ♀ — "E. B. Southwick, Type," both pins with a purple paper tab attached and the female with determination label "*Liburnia detecta* Van D." Macropterous.

Lectotype male, the above indicated specimen, here designated.

*Delphacodes foveata* (Van D.)

*Liburnia foveata* Van Duzee, Buffalo Soc. Nat. Sci. Bul. 5:257, 1897.

"New York, Ontario. Described from two male and eight female examples taken at Portage Falls, N. Y. May 30th, 1888 and Muskoka Lake, Ont., in July of the same year."

2 ♂ ♂, 3 ♀ ♀ — "Muskoka, Ont. July 1888, E. P. V. Coll., Type," on three pins as follows: 1 ♂; 2 ♀ ♀; 1 ♂, 1 ♀. The pin with both male and female has the determination label "*Liburnia foveata* Van D." Brachypterous.

Lectotype male, the above indicated specimen on pin by itself, here designated.

The following specimens are probably syntypes:

3 ♀ ♀ — "Muskoka, Ont., July 1888, E. P. V. Coll." Brachypterous.

1 ♀ — "Lancaster, N. Y. Aug 1886, E. P. V. Coll." Macropterous.

1 ♀ — "Lancaster, N. Y. May 1886, E. P. V. Coll." Brachypterous.

1 ♀ — "Clarence, N. Y. 9-4-92." Brachypterous.

*Delphacodes gillettei* (Van D.)

*Liburnia gillettei* Van Duzee, Buffalo Soc. Nat. Sci. Bul. 5:258, 1897.

"Colorado. Described from many examples received from Prof. C. P. Gillette including both the long and short winged forms of both sexes."



2 ♂♂, 2 ♀♀—"Colo. 633, Type," one brachypterous and one macropterous specimen of each sex, the brachypterous male with determination label "*Liburnia Gillettii* Van D."

Lectotype male, the above indicated brachypterous specimen, here designated.

The following specimens are probably syntypes:

3 ♀♀—"Colo. 633." Two brachypterous, 1 macropterous.

*Delphacodes incerta* (Van D.)

(Fig. 11)

*Liburnia* ? *incerta* Van Duzee, Buffalo Soc. Nat. Sci. Bul. 5:258, 1897.

"New York. Described from a single pair taken from a meadow near Buffalo on the 14th of June, 1893."

1 ♂—"Cheektowaga, N. Y. 6-14-93." Brachypterous.

Lectotype male, the above indicated specimen, here designated.

A second pin, bearing identical data, is without a specimen.

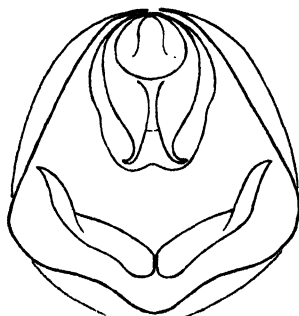


FIG. 11. *Delphacodes incerta* (Van Duzee). Male genitalia, full caudal view.

*Delphacodes kilmani* (Van D.)

*Liburnia kilmani* Van Duzee, Buffalo Soc. Nat. Sci. Bul. 5:253, 1897.

"New York. Described from one male and six female examples taken near Buffalo from June 18th to July 31st."

1 ♂—"Elma, N. Y. June 18th, '88, E. P. V. Coll., Type." Brachypterous.

1 ♀—"Buffalo, N. Y. E. P. V. Coll., Type." Macropterous.

2 ♀♀—"Colden N. Y. 7-31-89, Type," with determination label "*Liburnia Kilmani* Van D." in Van Duzee's handwriting. Brachypterous.

Lectotype male, the above indicated specimen, here designated.

*Delphacodes laminalis* (Van D.)

*Liburnia laminalis* Van Duzee, Buffalo Soc. Nat. Sci. Bul. 5:251, 1897.

*Liburnia lateralis* Van Duzee, Buffalo Soc. Nat. Sci. Bul. 5:253, 1897. New synonymy.

*Delphacodes laterana* Metcalf, General Catalogue Hemiptera 4(2):458, 1943. (New name for *Liburnia lateralis* Van Duzee, 1897, nec Fieber, 1879). New synonymy.

*Liburnia laminalis* was from "Mississippi. Described from one male and two female examples received from Mr. Howard Evarts Weed. The females were labeled 'Sept. 1892.'"

Van Duzee's *lateralis* was from "New York. Described from one male and three female examples taken at Lancaster on August 24th and September 10th, 1889. Another female was captured at Colden, N. Y., August 16, 1896."

The following syntypes of *laminalis* are at hand:

1 ♂—"Ag. Coll., Miss. Sept. '92., H. E. Weed., Type." Macropterous.

1 specimen, abdomen missing, presumably a female—"Miss., Type, 32." Macropterous.

Lectotype male, "Ag. Coll., Miss. Sept. '92, H. E. Weed., Type," here designated for *laminalis* Van Duzee.

Although the male specimen bears the date ascribed by Van Duzee to the females there seems no question that the above specimens are syntypes.

The following syntypes of *lateralis* are at hand:

1 ♂, 1 ♀, 1 specimen without abdomen—"Lancaster, N. Y. 9-10-89, E. P. V. Coll., Type," on one pin with determination label "*Liburnia lateralis* n. sp." Brachypterous.

Lectotype male, "Lancaster, N. Y. 9-10-89, E. P. V. Coll., Type," here designated for *lateralis* Van Duzee.

*Delphacodes lineatipes* (Van D.)

*Liburnia lineatipes* Van Duzee, Buffalo Soc. Nat. Sci. Bul. 5:255, 1897.

"New York, Ontario. Described from eight male and seven female examples taken near Muskoka Lake, Ont., about the first of August, 1888 and a single pair captured at Lancaster, N. Y., early in July."

4 ♂ ♂, 3 ♀ ♀—"Muskoka, Ont. July 1888, E. P. V. Coll., Type," on two pins, sexes separated, the pin with males bearing determination label "*Liburnia lineatipes* Van D." Brachypterous.

Lectotype male, lowermost of the above indicated specimens, here designated.

*Delphacodes lutulenta* (Van D.)

*Liburnia lutulenta* Van Duzee, Buffalo Soc. Nat. Sci. Bul. 5:252, 1897.

"New York. Described from six male and eight female examples, taken at Buffalo, May 4th to July 10th and Portage Falls, May 30th."

1 ♂, 1 ♀—"Portage N. Y. May 30th, 88, E. P. V. Coll., Type," the male with determination label "*Liburnia lutulenta* Van D." Brachypterous.

1 ♂, 2 ♀ ♀—"Buffalo, N. Y. 5-11-88, E. P. V. Coll., Type," male and one female on a single pin. Brachypterous.

Lectotype male, "Portage N. Y. May 30th, 88, E. P. V. Coll., Type," here designated.

The following specimens are probably syntypes:

2 ♂ ♂—"Portage N. Y. May 30th, 88, E. P. V. Coll." Brachypterous.

1 ♀—"Buffalo, N. Y. 5-11-88, E. P. V. Coll." Brachypterous.

1 ♀—"Buffalo, N. Y. 7-10-88, E. P. V. Coll." Brachypterous.

*Delphacodes oclusa* (Van D.)

*Liburnia oclusa* Van Duzee, Buffalo Soc. Nat. Sci. Bul. 5:256, 1897.

"Los Angeles, Calif. Described from one male and two female examples received from Mr. D. W. Coquillett (Nos. 191 and 192.)."

1 ♂—"192, California, Coquillett, Type." Brachypterous.

1 ♀—"California, Coquillett, Type," with determination label "*Liburnia oclusa* Van D." Brachypterous.

1 ♀—"191, Type." Macropterous.

Lectotype male, the above indicated specimen, here designated.

*Delphacodes puella* (Van D.)

*Liburnia puella* Van Duzee, Buffalo Soc. Nat. Sci. Bul. 5:250, 1897.

"New York, New Jersey, Mississippi, Iowa. Described from numerous individuals of both sexes."

2 ♂ ♂, 1 ♀—"Miss., Type," the female with determination label "*Liburnia puella* nsp." Macropterous.

1 ♀—"Ag. Coll., Miss., H. E. Weed, Type." Macropterous.

1 ♂—"Miss., Type," is a macropterous specimen of *Pissonotus piceus* Van Duzee.

Lectotype male, one of the above indicated specimens, here designated.

The following specimens are probably syntypes:

1 ♂, 1 ♀—"Ag. Coll., Miss., H. E. Weed." Macropterous.

1 ♂—"N. Brunsw., N. J. 7-20." Macropterous.

1 ♂—"Miss." Macropterous.

1 ♂—"Lancaster, N. Y. Aug. 1886, E. P. V. Coll." Macropterous.

*Euidella weedi* (Van D.)

*Liburnia weedi* Van Duzee, Buffalo Soc. Nat. Sci. Bul. 5:252, 1897.

"Mississippi. Described from a single male example received from Mr. Howard Evarts Weed."

1 ♂—"Miss., 30," with determination label "*Liburnia Weedi* Van D."

Holotype male, the above indicated specimen.

*Kelisia axialis* Van D.

Buffalo Soc. Nat. Sci. Bul. 5:232, 1897.

"Described from two examples representing both sexes, taken at Lancaster, N. Y. in August 1886."

1 ♂, 1 ♀—"Lancaster, N. Y. Aug. 1886, E. P. V. Coll., Type, E P Van Duzee Collector," on one pin with red label "Lectoholotype ♂, Lecto-allotype ♀, *Kelisia axialis* Van Duzee, R. H. B.," with determination label "*Kelisia axialis* Van D." Male genital structures dissected and contained in small vial attached to pin.

Lectotype male, the above indicated specimen, designated by Beamer (Kansas Ent. Soc. Jour. 18:102, 1945).

*Laccocera obesa* Van D.

*Laccocera ? obesa* Van Duzee, Buffalo Soc. Nat. Sci. Bul. 5:244, 1897.

"Colorado. Described from a single female specimen received from Prof. C. P. Gillette."

1 ♀—"Col., Ac. Cat. 42, 36, Type," with determination label "*Delphacinus? obesa* Van D."

Holotype female, the above indicated specimen.

*Laccocera vittipennis* Van D.

Buffalo Soc. Nat. Sci. Bul. 5:242, 1897.

"New Hampshire, Colorado. Described from three female examples taken on Mt. Washington by Mrs. Annie Trumble Slosson, and one pair from the mountains of North West Colorado, collected by C. P. Gillette."

2 ♀ ♀—"MT. WASH'N.," one with "Type" and determination label "*Delphacinus vittatipennis* Van D."

1 ♂, 1 ♀—"Colo. 564, Type," are *zonata* Van Duzee.

Lectotype female, the above indicated specimen with determination label attached, here designated.

*Laccocera zonata* Van D.

*Laccocera zonatus* Van Duzee, Buffalo Soc. Nat. Sci. Bul. 5:243, 1897.

"Colorado. Described from a single pair received from Prof. C. P. Gillette."

1 ♂—"Colo. 566, Type," with determination label "*Delphacinus zonatus* Van D."

Lectotype male, the above indicated specimen, here designated.

*Liburnia lateralis* Van Duzee, 1897 = *Delphacodes laminalis* (Van Duzee, 1897).

*Liburnia osborni* Van Duzee, 1897 = *Delphacodes campestris* (Van Duzee, 1897).

*Megamelus davisi* Van D.

(Fig. 12)

Buffalo Soc. Nat. Sci. Bul. 5:235, 1897.

"Michigan. Received from Mr. G. C. Davis . . . who reports it as abundant on water lilies."

6 ♂ ♂, 1 ♀—"Mich., Davis, June," one male brachypterous, remaining specimens macropterous, mounted on four pins.

Lectotype male, one of the above indicated specimens, uppermost on the pin, here designated.

Although without "type," "cotype," or determination labels, the above specimens are thought to be syntypes.

*Megamelus paleatus* (Van D.)

*Stenocranus paleatus* Van Duzee, Buffalo Soc. Nat. Sci. Bul. 5:232, 1897.

"Florida. Described from one female received from Mr. C. W. Johnson of Philadelphia."

1 ♀—"Fla."

Holotype female, the above indicated specimen.

*Phyllodinus nervatus* Van D.

*Phyllodinus nervata* Van Duzee, Buffalo Soc. Nat. Sci. Bul. 5:241, 1897.

"New York and Canada. Not uncommon near Buffalo on damp weedy meadows in June. Also taken in Welland Co., Ont., and at Muskoka Lake in July."

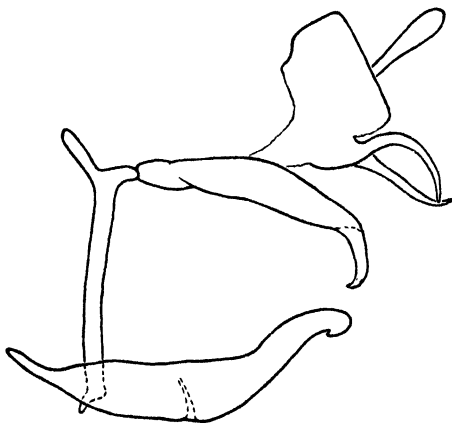


FIG. 12. *Megamelus davisi* Van Duzee. Male genitalia. Style, connective, aedeagus, and 10th segment, lateral view.

1 ♂—"Muskoka, Ont. July 1888, E. P. V. Coll., Type," with determination label "*Eurysa nervata* Van D."

2 ♀ ♀—"Elma, N. Y. June 18th, '88, E. P. V. Coll., Type."

Lectotype male, the above indicated specimen, here designated.

The following specimens are probably syntypes:

1 ♀—"Fort Erie, Ont. July 4th. 1887, E. P. V. Coll."

3 ♀ ♀—"Lancaster N. Y. June 11, 1887, E. P. V. Coll."

*Pissonotus aphidioides* Van D.

Buffalo Soc. Nat. Sci. Bul. 5:239, 1897.

"New York. Described from two female examples, one taken at Salamanca, August 2nd, 1889, the other at Colden a few days earlier."

1 ♀—"Salamanca, N. Y. 8-2-89, Type, E. P. Van Duzee, Collector." Brachypterous specimen.

1 ♀—"Colden, N. Y. 7-31-89, Type, E. P. Van Duzee, Collector," and

with determination label "*Pissonotus aphidiaides* [sic] Van D." Brachypterous specimen.

Lectotype female, "Salamanca, N. Y. 8-2-89, Type, E. P. Van Duzee, Collector," here designated.

*Pissonotus ater* Van D.

Buffalo Soc. Nat. Sci. Bul. 5:237, 1897.

"Near Buffalo, N. Y. Described from a single female example taken on Grand Island in Niagara River, Sept. 11th, 1892."

1 ♀—"Grand Isd., N. Y. 9-11-92, Type, E. P. Van Duzee, Collector," and with determination label "*Pissonotus ater* Van D." Brachypterous specimen.

Holotype female, the above indicated specimen, here designated.

*Pissonotus basalis* Van Duzee, 1897 = *Pissonotus marginatus* Van Duzee, 1897.

*Pissonotus brunneus* Van D.

Buffalo Soc. Nat. Sci. Bul. 5:239, 1897.

"New York. Described from six female examples, four taken near Buffalo in September, and two from New York City taken by Mr. E. B. Southwick in June."

3 ♀ ♀—"Grand Isd., N. Y. 9-11-92, Type, E. P. Van Duzee, Collector," mounted on one pin and with determination label "*Pissonotus brunneus* Van D." Brachypterous specimens.

1 ♀—"E. B. Southwick [on green paper], Type," and pin with a gray paper tab. Brachypterous specimen.

Lectotype female, uppermost specimen "Grand Isd., N. Y. 9-11-92, Type, E. P. Van Duzee Collector," here designated.

*Pissonotus delicatus* Van D.

Buffalo Soc. Nat. Sci. Bul. 5:237, 1897.

*Pissonotus pallipes* Van Duzee, Buffalo Soc. Nat. Sci. Bul. 5:238, 1897. New synonymy.

Van Duzee's *delicatus* was from "California. Described from a single female specimen received from Mr. D. W. Coquillett and captured near Los Angeles."

*P. pallipes* was from "California, Colorado. Described from one pair received from Prof. C. P. Gillette, taken among the mountains of North Western Colorado, and two females taken near Los Angeles, California, by Mr. D. W. Coquillett."

The following specimen of *delicatus* is at hand:

1 ♀—"658, Type," with determination label "*Pissonotus delicatus* Van D." Brachypterous specimen.

Holotype female, the above indicated specimen.

The following material of *pallipes* is at hand:

1 ♂—"Col., Ac. Cat 155, Type," with determination label "*Pissonotus pallipes* Van D." Two notes attached to pin, as follows: "Note. Base of

abdomen badly mutilated when red'd. from Ames, Iowa. The long wings covered the damaged section which was only seen when figuring external parts of genitalia. In removing specimen from stage of microscope the slight jar caused the abdomen to flip off the card point. I failed to recover it. Fig. of the pygofer and visible gen. is correct, however. W. M. G." and "Type ♂, (macropterous example) *Pissonotus pallipes* V. D., Pyg. and ext. gen. figd. before dissection." Macropterous specimen.

1 ♀—"Col., Ac. Cat 155, Type." Macropterous specimen.

1 ♀ (abdomen only)—"Calif. Type," with label "*pallipes*, Type." Macropterous specimen.

Lectotype male, the above indicated specimen, here designated for *pallipes* Van Duzee.

A critical study of the above specimens of *pallipes*, the holotype of *delicatus*, and many specimens from the western United States convinces me that *pallipes* is the macropterous form of *delicatus*. Accordingly, *pallipes* Van Duzee, 1897, is suppressed as a synonym of *delicatus* Van Duzee, 1897.

*Pissonotus dorsalis* Van D.

(Figs. 13, A, B, C, D)

Buffalo Soc. Nat. Sci. Bul. 5: 239, 1897.

"New York. Described from one pair captured in July, at Lancaster and Colden."

1 ♂—"Lancaster, N. Y. 7-12-89, E. P. V. Coll., Type, E. P. Van Duzee, Collector," with determination label *Pissonotus dorsalis* Van D." and note "Type ♂—Figd, *Piss. dorsalis*, Ames, Iowa, Coll." Genital structures dissected and mounted between glass cover slips in hole punched in quadrangular card attached to pin and with marginal notes "♂ gen. W. M. G. 1928, *Pissonotus dorsalis* Van D., Type ♂." Brachypterous specimen.

1 ♀—"Colden, N. Y. 7-31-89, Type, E. P. Van Duzee, Collector," and with determination label "*P. dorsalis* Van D., ♀, det. Van D." Brachypterous specimen.

Lectotype male, the above indicated specimen, here designated.

*Pissonotus marginatus* Van D.

(Figs. 13, E, F, G, H)

Buffalo Soc. Nat. Sci. Bul. 5: 236, 1897.

*Pissonotus basalis* Van Duzee, Buffalo Soc. Nat. Sci. Bul. 5: 238, 1897. New synonymy.

Van Duzee's *marginatus* was from "Lancaster, N. Y. July and Sept. Described from a single pair. More recently, June 1896, I took a fine female of this species at Hamburg, N. Y."

*P. basalis* was "Described from one female taken at Lancaster, N. Y., July 4th, 1888."

The following material of *marginatus* is at hand:

1 ♂ (genitalia only)—"Lancaster, N. Y. 9-3-88, E. P. V. Coll., Type, E. P. Van Duzee, Collector," with determination label "*Pissonotus marginatus* Van D." at the bottom of which is written "Received in damaged

condition." The pin also bears the notation, presumably referring to drawings of the genitalic characters "Figd. —d, Gen. diss. W M G [W. M. Giffard]." The genital structures have been dissected and mounted between glass cover slips in a hole punched in a quadrangular card attached to the pin and with the marginal notations " $\delta$  Gen. W. M. G. 1928, *Pissonotus marginatus* V. D., Type  $\delta$ ."

1 ♀—"Lancaster, N. Y. 7-12-89, E. P. V. Coll., Type, E. P. Van Duzee, Collector," with determination label "*P. marginatus* V. D., ♀, det. Van Duzee." Brachypterous specimen.

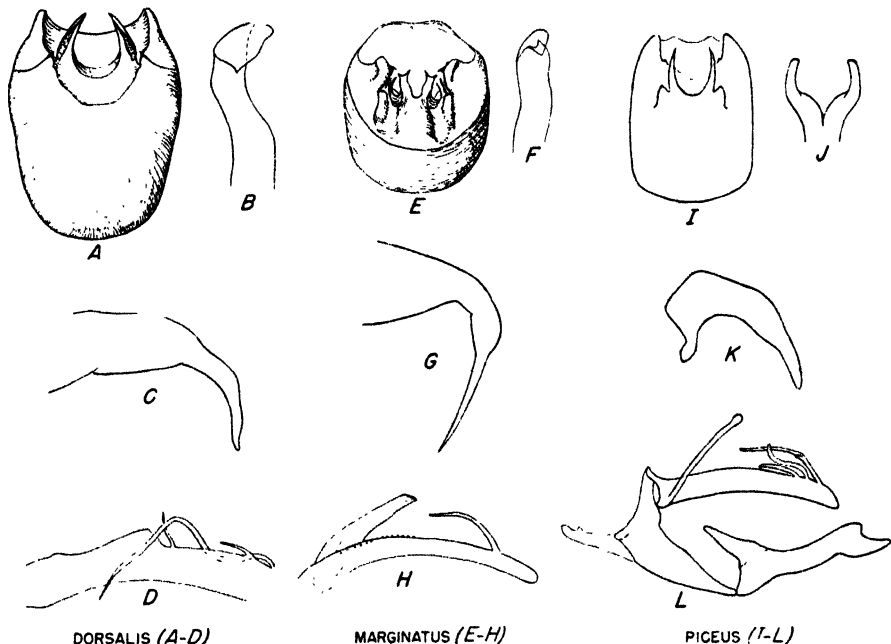


FIG. 13. *Pissonotus*. Male genitalia. A, genital capsule, ventral view; B, left style, ventral view; C, hook of 10th segment, lateral view; and D, aedeagus (tip missing), lateral view, of *dorsalis* Van Duzee. E, genital capsule, caudo-ventral view; F, left style, ventral view; G, hook of 10th segment, lateral view; and H, aedeagus, lateral view, of *marginatus* Van Duzee. I, genital capsule, ventral view; J, distal portion of styles, ventral view; K, 10th segment, lateral view; and L, style, connective, and aedeagus, lateral view, of *piceus* (Van Duzee). Genital capsules shown at one-half the scale of other drawings.

Lectotype male, remaining parts of the above indicated specimen, here designated for *marginatus* Van Duzee.

The following specimen of *basalis* is at hand:

1 ♀—"Lancaster, N. Y. 7-4-88, Type, E. P. Van Duzee, Collector," and with determination label "*Pissonotus basalis* Van D." Macropterous specimen.

Holotype female, the above indicated specimen.

Critical comparison of the holotype of *basalis* with the brachypterous female syntype of *marginatus* convinces me that they represent but a



single species. Accordingly, *basalis* Van Duzee, 1897, is suppressed as a synonym of *marginatus* Van Duzee, 1897.

*Pissonotus pallipes* Van Duzee, 1897 = *Pissonotus delicatus* Van Duzee, 1897.

*Pissonotus piceus* (Van D.), new combination

(Figs. 13, I, J, K, L)

*Megamelus piceus* Van Duzee, Michigan Agr. Expt. Sta. Bul. 102: 27 and 28, [1893] 1894.

"New York and Michigan. Described from many examples of both sexes taken in western New York on grass in low swampy meadows in August and September, and one female taken on celery at Kalamazoo, Mich., August 26, 1893, by Mr. G. C. Davis."

1 ♂, 1 ♀ — "Clarence, N. Y. 9-4-92, Type, E. P. Van Duzee, Collector," mounted on one pin. Brachypterous specimens. Male with genital capsule cleared and contained in a small vial attached to pin.

1 ♀ — "Lancaster, N. Y. Aug. 1886, E. P. V. Coll., Type, E. P. Van Duzee, Collector," with determination label "*Megamelus piceus* Van D." Macropterous specimen.

Lectotype male, the above indicated specimen, here designated.

The following specimens are probably syntypes also:

1 ♂ — "Grand Isd., N. Y. 9-11-92." Macropterous.

2 ♀ ♀ — "Clarence, N. Y. 9-4-92." Macropterous and brachypterous.

*Prokelisia crocea* (Van D.), new combination

*Kelisia crocea* Van Duzee, Buffalo Soc. Nat. Sci. Bul. 5: 233, 1897.

"Iowa. Described from 5 ♂ and 3 ♀ examples received from Prof. Herbert Osborn labeled 'Ames, Iowa.'"

1 ♂ — "Osborn, Ames, Ia., Type," with determination label "*Stenocranus croceus* Van D." in Van Duzee's handwriting, a red label "Cotype, "*Kelisia crocea* Van D., R. H. B." and a red label "Lechtholotype, *Liburnia crocea* (V. D.), R. H. B." Genital structures dissected and contained in a small vial attached to pin.

2 ♀ ♀ — "Osborn, Ames, Ia., Type."

Lectotype male, the above indicated specimen, designated by Beamer (Kansas Ent. Soc. Jour. 18: 100, 1945).

The following specimens are probably syntypes:

1 ♂, 1 ♀ — "Osborn, Ames, Ia."

Beamer (Kansas Ent. Soc. Jour. 18: 100, 1945; 19: 83, 1946) referred *crocea* to *Liburnia* Stal, 1866, but that name has been suppressed as an absolute synonym of *Embolophora* Stal, 1853 (see China, Ann. Mag. Nat. Hist. (11) 4: 582-584, 1939). *Crocea* is not congeneric with the South African *monoceros* Stal, type of *Embolophora*, but appears to be properly placed in *Prokelisia* Osborn, type *setigera* Osborn, 1905.

*Prokelisia marginata* (Van D.)

*Megamelus marginatus* Van Duzee, Buffalo Soc. Nat. Sci. Bul. 5: 234, 1897.

"New York and New Jersey. Described from several examples taken by Prof. J. B. Smith at Anglesea, N. J., May 28th, and near New

York City and at Ravenswood, N. Y., Aug. 28th, 1890, by Mr. E. B. Southwick."

3 ♂♂, 3 ♀♀—"Anglesea, N. J. 5-28," one male and two females with "Type" and the same male with determination label "*Stenocranus marginatus* Van D."

1 ♂—"Ravenswood, N. Y., 8-28-1890, E. B. Southwick."

1 ♀—"E. B. Southwick," with purple paper tab on pin.

Lectotype male, "Anglesea, N. J. 5-28, Type," here designated.

*Stenocranus lautus* Van D.

Buffalo Soc. Nat. Sci. Bul. 5: 231, 1897.

"New York, Virginia. Described from two male examples; One received from Dr. E. B. Southwick, taken near New York City; the other from the National Museum labeled 'Virginia, Oct. 9th, 1881.'"

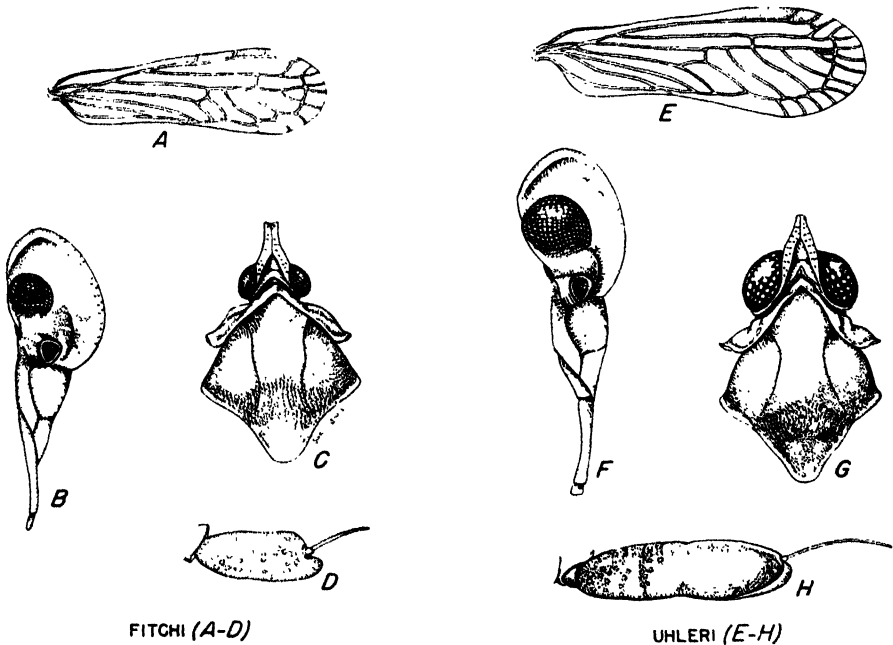


FIG. 14. *Amalopta*. A, forewing; B, head, lateral view; C, head and thorax, dorsal view; and D, antenna, caudal view, of *fitchi* Van Duzee. E, forewing; F, head, lateral view; G, head and thorax, dorsal view; and H, antenna, caudal view, of *uhleri* Van Duzee.

1 ♂—"E. B. Southwick [on green paper]," with purple tab on pin, with red label "Lectotype, *S. lautus* V. D., R. H. B.," and with determination label "*Stenocranus lautus* V. D. (= *vittatus* Stal)." Genital structures dissected and contained in small vial attached to pin.

Lectotype male, the above indicated specimen, designated by Beamer (Kansas Ent. Soc. Jour. 19: 7, 1946).

## FAMILY DERBIDAE

*Amalopota fitchi* Van D.

(Figs. 14, A, B, C, D)

*Amalopota Fitchi* Van Duzee, Canad. Ent. 25:280, 1893.

"New York. Described from a single example beaten by me from a tree of the wild black cherry among the hills about twenty miles south of this city [Buffalo], on July 28th, 1892."

1 ♀—"Colden, N. Y. 8-28-92, Type," with determination label "*Amalopota Fitchi* Van D."

Holotype female, the above indicated specimen.

*Amalopota uhleri* Van D.

(Fig. 14, E, F, G, H)

*Amalopota Uhleri* Van Duzee, Canad. Ent. 21:178, 1889.

"Described from five individuals—a pair taken *in coitu* Sept. 3rd, 1888, two females taken the same day, and another female taken by W. J. Palmer, Jr., of this city, a week later, all at Lancaster, N. Y."

1 ♂, 2 ♀ ♀—"Lancaster, N. Y. 9-3-88, E. P. V. Coll., Type," the male and one female *in coitu* on one pin, the other female on pin bearing determination label "*Amalopota Uhleri* Van Duzee."

Lectotype male, the above indicated specimen, here designated.

*Cedusa californica* (Van D.)*Lamenia Californica* Van Duzee, Canad. Ent. 23:169, 1891.

"Los Angeles, California. Described from six examples, all males, received from Mr. D. W. Coquillett. (Nos. 642 and 643)."

1 ♂—"Calif., Coquill., Type." No "cotype" label.

1 ♀—"Calif., Coquill., Type," with determination label "*Lamenia californica* Van D."

Lectotype male, the above indicated specimen, here designated.

The female indicated above is believed to be a syntype even though the original material was stated to be all males, since the sex of specimens in this group was occasionally confused by Van Duzee.

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## FAMILY CERCOPIDAE

<i>Aphrophora canadensis</i> Walley	4♂♂, 4♀♀—paratypes
<i>Aphrophora princeps</i> Walley	2♂♂ —paratypes

## FAMILY MEMBRACIDAE

<i>Heliria gemma</i> Ball	1♀—paratype
<i>Telamona gibbera</i> Ball	1♂ —paratype

## FAMILY CICADELLIDAE

<i>Alapus attenuatus</i> Lawson	1♀—paratype
<i>Deltocephalus vanduzeei</i> Gillette and Baker	1♀—syntype
<i>Deltocephalus yavapai</i> Tuthill	1♂, 1♀—paratypes
<i>Dikraneura cockerelli</i> Gillette	1♀—syntype
<i>Dikraneura cruentata</i> Gillette	1♂, 1♀—syntypes
<i>Driotura vittata</i> var. <i>nigra</i> Lawson	1♀—paratype
<i>Empoasca atrolabes</i> Gillette	2♀♀—syntypes
<i>Erythroneura pyra</i> McAtee	1♂ —holotype
<i>Erythroneura rubroscuta</i> (Gillette)	2♀♀—syntypes
<i>Eutettix acutus</i> Hepner	1♂ —paratype
<i>Eutettix aequalis</i> Hepner	1♂ —paratype
<i>Eutettix apicalis</i> Hepner	1♀—paratype
<i>Eutettix grandis</i> Hepner	1♂ —paratype
<i>Eutettix latus</i> Hepner	2♀♀—paratypes
<i>Eutettix minutus</i> Hepner	1♂ —paratype
<i>Eutettix pediculus</i> Hepner	1 (abdomen missing)—paratype
<i>Eutettix planus</i> Hepner	1♂ —paratype
<i>Eutettix prinoides</i> Hepner	1♂, 1♀—paratypes
<i>Eutettix querci</i> var. <i>albus</i> Hepner	1♂, 1♀—paratypes
<i>Eutettix rugosus</i> Hepner	1♂ —paratype
<i>Eutettix subspinosus</i> Hepner	1♂ —paratype
<i>Eutettix variabilis</i> Hepner	1♂ —paratype
<i>Gillettiella labiata</i> (Gillette)	2♂♂, 3♀♀—syntypes
<i>Latalus curtus</i> Beamer and Tuthill	1♂, 1♀—paratypes
<i>Latalus mundus</i> Beamer and Tuthill	1♂, 1♀—paratypes
<i>Lonatura punctifrons</i> Beamer	1♂, 1♀—paratypes
<i>Laevicephalus bimaculatus</i> (Gillette and Baker)	2♂♂ —syntypes
<i>Laevicephalus cookei</i> (Gillette)	1♂, 1 (abdomen missing)—syntypes
<i>Laevicephalus parvulus</i> (Gillette)	1♂, 1♀—syntypes
<i>Macropsis osborni</i> Breakey	2♀♀—paratypes
<i>Osbornellus rotundus</i> Beamer	3♂♂, 1♀—paratypes
<i>Pasadenus omani</i> Ball	1♂ —paratype
<i>Polyamia incerta</i> Beamer and Tuthill	1♂, 1♀—paratypes
<i>Stirellus dixianus</i> var. <i>robustus</i> Thomas	1♂, 1♀—paratypes
<i>Stragania robusta</i> (Uhler)	1 (abdomen missing)—syntype
<i>Typhlocyba cymba</i> var. <i>unipuncta</i> McAtee	1♀—paratype
<i>Typhlocyba gillettei</i> var. <i>apicata</i> McAtee	2♂♂ —paratypes
<i>Typhlocyba gillettei</i> var. <i>fitchii</i> McAtee	1♂ —paratype
<i>Typhlocyba gillettei</i> var. <i>sincera</i> McAtee	3♀♀—holotype and paratypes
<i>Typhlocyba niobe</i> McAtee	1♂, 2♀♀—allotype♀ and paratypes
<i>Typhlocyba phryne</i> var. <i>subpulchra</i> McAtee	1♀—holotype
<i>Typhlocyba piscator</i> McAtee	1♂, 1♀—allotype♀ and paratype
<i>Typhlocyba pomaria</i> McAtee	1♂ —paratype
<i>Typhlocyba unca</i> McAtee	3♀♀—allotype and paratypes



**SUPERFAMILY FULGOROIDEA****FAMILY DELPHACIDAE**

<i>Phyllodinus tessellatus</i> Ball .....	1♂	—paratype
<i>Stenocranus delicatus</i> Beamer .....	2♂♂, 2♀♀	—paratypes
<i>Stenocranus pallidus</i> Beamer .....	1♂, 3♀♀	—paratypes

# APOCYNUM IN IOWA<sup>1</sup>

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Experiment Station, Ames, Iowa*

Received October 1, 1946

*Apocynum*, the only genus of the Apocynaceae present in the state, has been the object of renewed economic interest. Recently the plants were investigated as a possible source of fiber and rubber. It was necessary to know what species were present in the state and to note several name changes. Within the genus the concept of the limitation of species and varieties has presented many problems. The presence of intergrading forms and probably hybridization are among those which have added to the difficulties of classification. The combination of these facts, coupled with the monograph of this family by Dr. R. E. Woodson (5) that forms the basis of this paper, led to this investigation of the genus.

Cratty's (3) catalogue of Iowa plants lists five species: *A. cannabinum* L., *A. pubescens* R. Br., *A. sibiricum* Jacq., *A. androsaemifolium* L., and *A. medium* Greene. Three species, three varieties, one hybrid, and one secondary hybrid have been recognized in this paper. Briefly the changes are as follows: *A. cannabinum* has been broken up into three entities, all of which are present in the state; *A. cannabinum* L., *A. cannabinum* L. var. *pubescens* (R. Br.) A. DC., and *A. cannabinum* L. var. *glaberrimum* A. DC. In addition to *A. sibiricum* a variety has been recognized, *A. sibiricum* Jacq. var. *cordigerum* (Greene) Fern. *A. androsaemifolium* remains unchanged. *A. medium* is considered a hybrid. A secondary hybrid, variety *leuconeuron* of *A. medium*, has been added.

*A. medium* is considered by Dr. Anderson (1) to be a convenient way to designate this hybrid. However, he does not give it specific standing with *A. androsaemifolium* and *A. cannabinum*. Too great a variability among the progeny and a high degree of pollen sterility precludes it being a good species. *A. medium* is not considered as a species in this paper.

Distribution maps have been made showing known collections in the state. (Figs. 1-6.) The specimens studied are in the Iowa State College, University of Iowa, and Parsons College herbaria.

Following is a key to the Iowa species, brief descriptions, and a few citations of specimens. To Number 2 and Number 3 are relegated the intergrading forms difficult to segregate, which are assumed to be primary or secondary hybrids between *A. androsaemifolium* and *A. cannabinum*. The follicle was too infrequent in Iowa material to be used

<sup>1</sup> Journal Paper No. J-1400 from the Botany and Plant Pathology Section of the Iowa Agricultural Experiment Station, Ames, Iowa. Project No. 86.

FIG. 1.

- *Apocynum androsaemifolium*

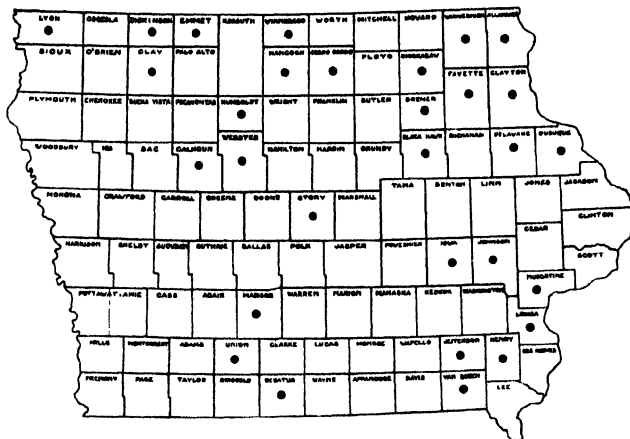


FIG. 2.

- *Apocynum medium*
- ▲ *Apocynum medium* var. *leuconeuron*

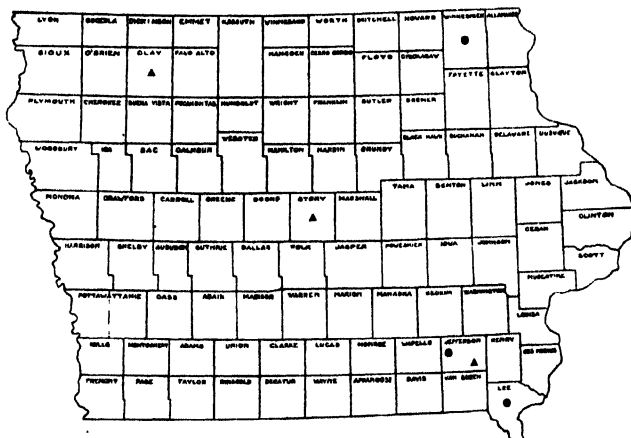


FIG. 3.

- *Apocynum cannabinum*
- △ *Apocynum cannabinum* var. *pubescens*

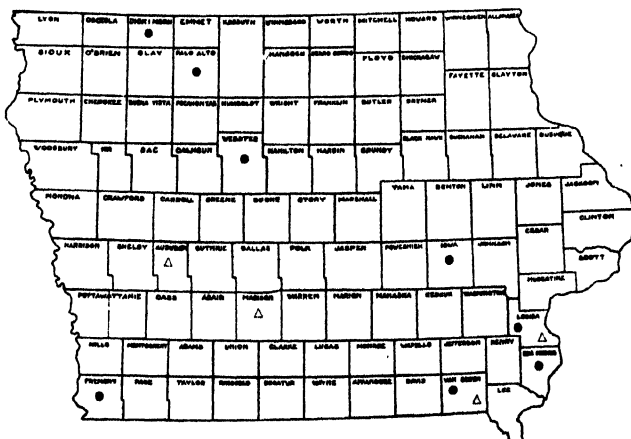


FIG. 4.

- *Apocynum cannabinum* var. *glaberrimum*

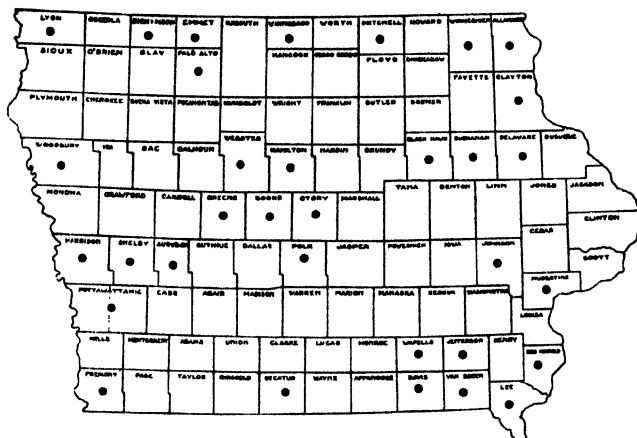


FIG. 5.

- *Apocynum sibiricum*

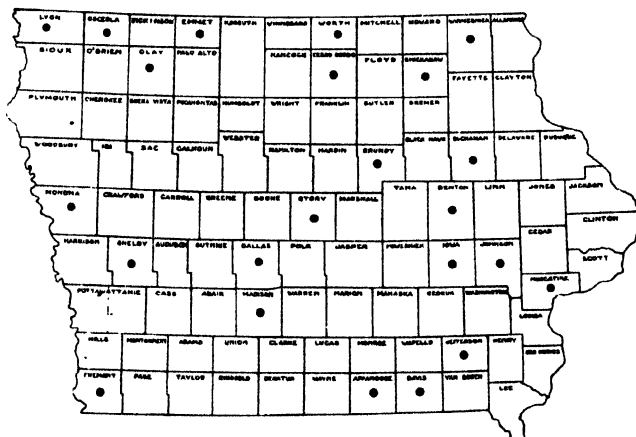
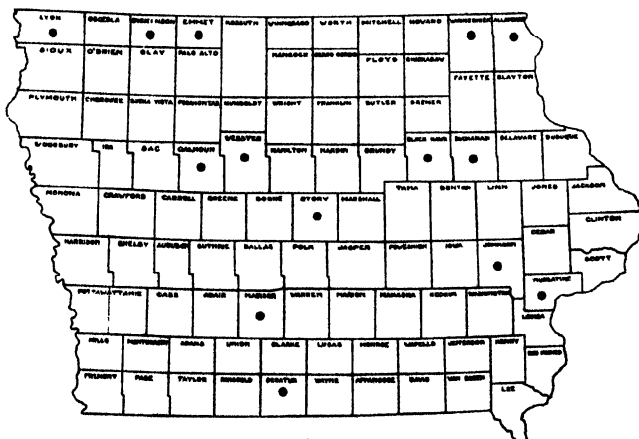


FIG. 6.

- *Apocynum sibiricum* var. *cordigerum*



in the key. The drooping, spreading or ascending character of the leaves can be used with certainty only in the field.

### KEY TO APOCYNUM IN IOWA

- a. Corolla 2 to 3 or more x the length of the calyx.
  - b. Corolla at least 3 x the length of the calyx; branching mostly alternate, often dichotomous; leaves usually ovate, pubescent on the lower surface, drooping; inflorescence an irregularly branched, few-flowered cyme, axillary flowers commonly present.
    - 1. *Apocynum androsaemifolium*
  - bb. Corolla at least 2 x the length of the calyx; branching alternate, opposite or sub-opposite; leaves ovate to oblong-lanceolate, pubescent to tomentulose to glabrous, spreading; inflorescence a few to many flowered cyme, axillary flowers occasionally present.
    - c. Leaves pubescent to tomentulose beneath.
      - 2. x *Apocynum medium* (a hybrid)
    - cc. Leaves glabrous.
      - 3. x *Apocynum medium* var. *leuconeuron*  
(a secondary hybrid)
  - aa. Corolla 1½ to 2 x the length of the calyx; branching usually opposite to sub-opposite; leaves ovate, oblong, or lanceolate, spreading to ascending; inflorescence a regularly branched, paniculate to corymbose, densely flowered cyme, no axillary flowers.
    - d. Leaves all petiolate; upper leaves slightly smaller than the lower leaves.
      - e. Leaves glabrous on the upper surface.
        - f. Leaves pubescent on the lower surface.
          - 4. *Apocynum cannabinum*
        - ff. Leaves glabrous on the lower surface.
          - 5. *Apocynum cannabinum* var. *glaberrimum*
      - ee. Leaves pubescent on both surfaces.
        - 6. *Apocynum cannabinum* var. *pubescens*
    - dd. Leaves (at least some of them) sessile; upper leaves noticeably smaller than the lower leaves.
      - g. Lowermost leaves sessile, sometimes auricled, lanceolate to oblong-lanceolate not tapering at the base; middle and upper leaves with petioles, lanceolate to elliptic-lanceolate, tapering at the base; angles of branches 45° or less.
        - 7. *Apocynum sibiricum*
      - gg. Lower and middle leaves sessile, deeply cordate, amplexicaul, ovate; uppermost leaves sessile or nearly so, oval to oblong-lanceolate; angles of branches 50° to 90°.
        - 8. *Apocynum sibiricum* var. *cordigerum*
- 1. *Apocynum androsaemifolium* L. Sp. Pl. ed. 2, 311. 1762.  
*Apocynum androsaemifolium* L. var. *incanum* A. DC. in DC. Prodr. 8: 439. 1844.

Corolla campanulate, sometimes pink, length 5 to 8 mm.; leaves glabrous on the upper surface, pubescent on the lower surface; entire plant loosely branched and spreading; follicle straight, 10–15 cm. in length; seed 2 mm. long, coma 2 cm. long, tawny-colored.

Open wooded slopes along the Little Sioux River, Gillet Grove, Clay Co., June 27, 1936, *Ada Hayden* 10,274; Fayette Co., July, 1893, *Bruce Fink*.

2. x *Apocynum medium* Greene Pittonia 3:229. 1897.

(*Apocynum cannabinum* L. x *A. androsaemifolium* L.)

A highly variable entity; leaves glabrous on the upper surface, various degrees of pubescence on the lower surface.

3. x *Apocynum medium* Greene var. *leuconeuron* (Greene) Woodson. Ann. Mo. Bot. Gar. 17:112. 1930.

*Apocynum leuconeuron* Green. Leaf. Bot. Obs. and Crit. 2:178. 1912. (*Apocynum cannabinum* x *A. androsaemifolium*)

A highly variable entity; leaves glabrous; a preponderance of characters of the parent *A. cannabinum*.

4. *Apocynum cannabinum* L. Sp. Pl. 213. 1753.

Same as the following except for the pubescence on the underside of the leaves.

5. *Apocynum cannabinum* L. var. *glaberrimum* A. DC. in DC Prodr. 8:439. 1844.

Corolla sphaerico-cylindrical, 2.5 to 3 mm. in length, 2.5 mm. broad, upper leaves slightly smaller than the lower leaves and lanceolate; lower leaves both lanceolate and ovate; petioles on the average 3 to 4 mm. in length, occasionally a few of the lower leaves may be sessile; entire plant bushy, both branches developing at a node, angle of the branches wider than either *A. sibiricum* or its variety; fruit usually falcate, pendulous, 12 to 20 cm. in length; seed 4 to 5 mm. in length, coma 2.5 to 3 cm. in length, white. (Fig. 7.)

Wet soil around standing water, Palo Alto Co., July 8, 1940, *Ada Hayden* 7108; Border of Woods, Fayette, Fayette Co., Aug. 1894. *Bruce Fink*.

6. *Apocynum cannabinum* L. var. *pubescens* (R. Br.) A. DC. in DC. Prodr. 8:440. 1844.

Inflorescence pubescent; leaves variable, broadly ovate, lanceolate, oblong and even slightly obovate; stem pubescent to glabrous.

Wet roadside depression in open country near virgin prairie, Palo Alto Co., Sept. 21, 1942, *Ada Hayden* (ver. R. E. Woodson) 7532.

7. *Apocynum sibiricum* Jacq. Hort. Vindob. 3:37. 1770.

*Apocynum hypericifolium* Ait. Hort. Kew 1:304. 1789.

*Apocynum cannabinum* L. var. *hypericifolium* A. Gray Man. 365. 1848.

Corolla sphaerico-cylindrical, average length 2 to 3 mm.; Lowermost leaves sometimes clasping and slightly auricled; upper leaves with petioles 2 to 4 mm. in length; branches opposite but only one branch developing at a node, hence a more upright and slenderer plant than *A. cannabinum* and its varieties; fruit divergent, 5 to 8 cm. long; seed 4 mm. long, coma 10 mm. long. (Figs. 8 and 10.)

Sloughs in Dewey's Pasture, Ruthven, Clay Co., Aug. 17, 1942, *Ada Hayden* (ver. R. E. Woodson) No. 7533; High Lake, Emmet Co., June 29, 1923, *B. O. Wolden* 832; Field and waste places, Decatur Co., July 8, 1897, *T. J. Fitzpatrick* and *M. F. L. Fitzpatrick*.



FIG. 7. *Apocynum cannabinum* var. *glaberrimum*.

FIG. 8. *Apocynum sibiricum*.

FIG. 9. *Apocynum sibiricum* var. *cordigerum*.

8. *Apocynum sibiricum* Jacq. var. *cordigerum* (Greene) Fernald. *Rhodora* 37:328. 1935.

*Apocynum hypericifolium* Ait. var. *cordigerum* Beg. and Bel. Mem. Accod. Lincei V. 9:704. 1913.

*Apocynum cordigerum* Greene. Leaflets 2:164. 1911.

Uppermost leaves sessile or with petioles 1.5 to 2 mm. in length. Leaves more ascending, thicker, and with a softer texture than in *A. sibiricum*; branching opposite but only one branch developing at a node, however the angles of the branches are wider than in *A. sibiricum*, hence a more sprawling plant. (Figs. 9 and 10.)

Fields and waste places, common, Decatur Co., July 8, 1897, *T. J. Fitzpatrick* and *M. F. L. Fitzpatrick*; Dolliver, Emmett Co., July 6, 1926, *L. H. Pammel* 235.

Thanks is due to Dr. G. J. Goodman who suggested the problem and gave helpful suggestions. The writer also wishes to express her appreciation to Dr. R. E. Woodson for critical comments.

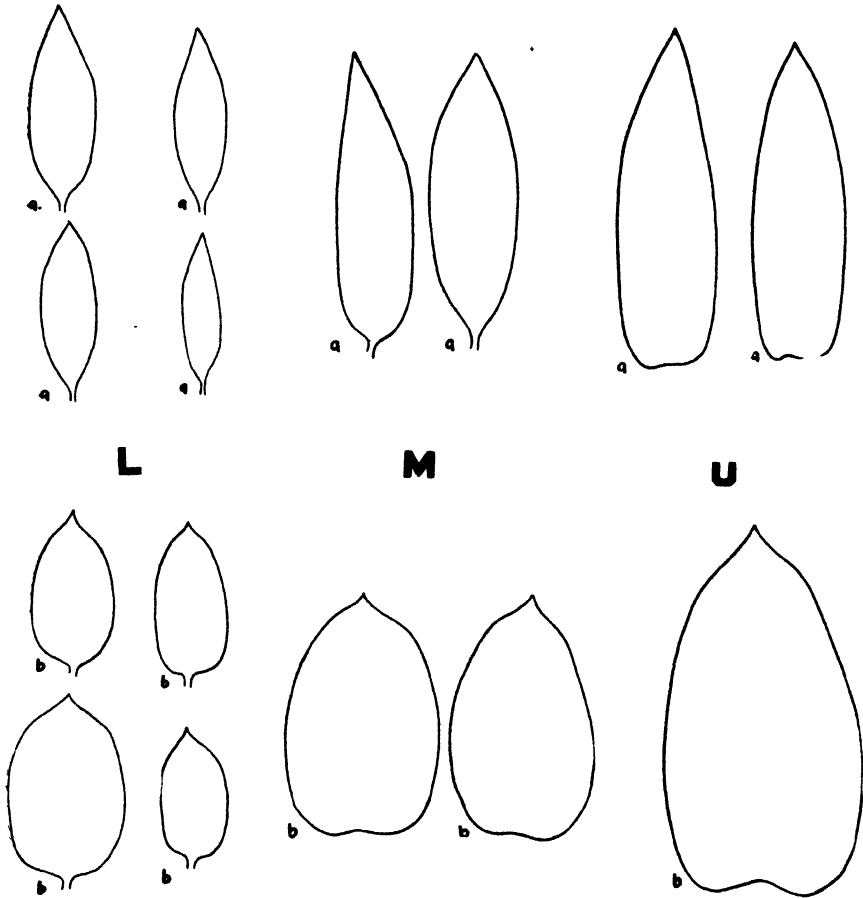


FIG. 10. L—Lower leaves; M—Middle leaves; U—Upper leaves.  
 a. *Apocynum sibiricum*  
 b. *Apocynum sibiricum* var. *cordigerum*

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# THE EFFECT OF PANTOTHENATE DEFICIENCY ON TRYPANOSOMA LEWISI INFECTION IN THE RAT

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It has been reported by Becker, Manresa, and Johnson (1943) that the multiplicative period of *Trypanosoma lewisi* in the rat's blood was inordinately lengthened by feeding the host a ration deficient in pantothenic acid, and that as a result there was an exaltation of parasite density, sometimes resulting in death of the host. Because Bartonella-like bodies were frequently encountered in stained blood films from the affected rats, and blood picture and spleen weight were suggestive of bartonellosis, there was no assurance that the altered course of the infection was not due primarily to intercurrent infection rather than to the deficiency itself. In a reinvestigation of the effect of pantothenic acid deficiency on the course of *T. lewisi* infection it has been possible to eliminate the factor of intercurrent infection. In the new work the deficiency was effected by excluding the vitamin from the construction of the ration instead of by dry-heating as before. Observations were also made on the effect of the parasitemia on weight gains and on red and white cell counts.

## MATERIAL AND METHODS

Cultures of the strain of *Trypanosoma lewisi* employed were obtained from the Army Medical School, Washington, D. C. The strain was supposed to be the one obtained from the senior author in 1943, then carried on in rats at that place. The strain at first exhibited greater multiplicative powers than hitherto, for counts per cubic millimeter of blood often exceeded those obtained before 1943. Bartonella did not appear in any of the pantothenate-deficient rats inoculated with trypanosomes in the blood of the rats seeded from the cultures, nor in any of the rats through which the trypanosomes were subsequently passed. The loss of this microorganism was possibly due to its inability to grow in the blood agar culture medium.

The host employed was the Wistar A rat. The colony is louse-free. One litter was used in each experiment. The rats were started on the test rations when the mean weights of the litters averaged from 59 to 79 grams (see parentheses under experiment numbers in Table 1). Rats of these weights did not show the extreme symptoms of pantothenic acid

<sup>1</sup> This investigation was supported in part by a grant from the Science Research Fund of Iowa State College.

TABLE 1

WEIGHT INCREASES AND TRYPANOSOME NUMBERS PER CUBIC MILLIMETERS OF BLOOD FOR RECIPIENTS AND NONRECIPIENTS OF PANTOTHENATE (P.A.)

Experiment Number	Rat No.	Sex	P.A.	Weight Increases (grams) *						Trypanosome Numbers (10,000) †				
				P	7D	10D	12D	15D	17D	7D	10D	12D	15D	16D
1..... (73 grams)	1	M	—	36	....	68	69	....	....	....	11	7	....	....
	2	M	—	50	....	72	69	....	....	....	187	435	....	....
	3	F	—	30	....	55	51	....	....	....	2	2	....	....
	4	F	—	39	....	62	64	....	....	....	76	70	....	....
	5	M	+	44	....	109	119	....	....	....	53	67	....	....
	6	F	+	42	....	86	94	....	....	....	14	10	....	....
	7	F	+	39	....	69	77	....	....	....	3	0	....	....
	8	M	+	54	....	120	126	....	....	....	0	0	....	....
2..... (59 grams)	9	F	—	39	44	42	45	....	....	....	31	21	....	....
	10	M	—	45	58	58	57	....	....	....	174	141	....	....
	11	F	+	51	75	80	94	....	....	....	32	24	....	....
	12	M	+	58	103	120	134	....	....	....	2	1	....	....
	13	F	+	51	77	83	94	....	....	....	22	20	....	....
3..... (62.5 grams)	14	M	—	16	27	23	29	29	....	....	106	89	126	....
	15	M	—	26	32	38	35	26	....	....	111	84	118	....
	16	F	—	30	38	42	45	47	....	....	81	79	158	....
	17	F	+	57	91	97	93	107	....	....	6	2	1	....
	18	F	+	53	75	86	97	104	....	....	34	25	11	....
	19	M	+	65	109	127	143	157	....	....	5	5	4	....
4..... (70.5 grams)	20	M	—	39	54	61	65	67	....	....	80	95	52	....
	21	M	—	36	57	66	73	74	....	....	66	64	30	....
	22	F	—	37	61	64	73	70	....	....	23	21	19	....
	23	F	—	39	52	58	67	65	....	....	19	19	15	....
	24	M	+	62	97	96	....	....	....	....	9	12	4	....
	25	M	+	68	108	103	....	....	....	....	0	0	0	....
	26	F	+	52	83	81	....	....	....	....	0	0	0	....
	27	F	+	54	85	87	....	....	....	....	15	15	8	....
5..... (67.4 grams)	28	F	—	40	42	54	52	....	50	2	17	13	....	4
	29	M	—	41	44	48	51	....	50	10	48	27	....	17
	30	M	—	47	46	52	53	....	56	4	41	25	....	13
	31	F	—	38	40	49	50	....	50	8	19	20	....	8
	32	F	+	42	56	63	70	....	79	3	6	3	....	3
	33	F	+	44	62	70	80	....	89	3	4	1	....	1
	34	M	+	40	53	60	67	....	75	4	4	1	....	1
	A	F	—	37	55	66	76	....	82	Not inoculated				
	B	M	—	41	48	58	61	....	63	Not inoculated				
	C	F	—	37	42	53	56	....	58	Not inoculated				
	D	M	—	45	57	68	73	....	75	Not inoculated				
6..... (79 grams)	35	M	—	53	67	76	....	....	....	7	13	....	6	....
	36	F	—	41	52	62	....	....	....	13	17	....	1	....
	37	F	—	30	39	38	....	....	....	13	30	....	18	....
	38	M	—	49	57	65	....	....	....	15	5	....	3	....
	39	F	+	43	57	80	....	....	....	1	+	....	0	....
	40	F	+	47	61	67	....	....	....	+	+	....	0	....
	41	F	+	43	62	71	....	....	....	8	9	....	6	....
	42	M	+	57	79	89	....	....	....	+	+	....	+	....

\* P = Preparation Period (12-14 days)

7D = Seventh Day of Infection

10D = Tenth Day of Infection

12D = Twelfth Day of Infection, etc.

† Less than 5,000 indicated by +.

deficiency obtainable in younger rats, but nevertheless the course of the *T. lewisi* infection was affected. The experimental rats were inoculated intraperitoneally with about 100,000 trypanosomes each.

The deficient diet had the following composition: sucrose, 640 g.; vitamin-free casein, 220 g.; hydrogenated vegetable oil, 80 g.; cod liver oil, 20 g.; salt mixture, 40 g. The following amounts of crystalline vitamins were added to each 1,000 g. of the above: thiamine hydrochloride, 8 mg.; pyridoxine hydrochloride, 10 mg.; riboflavin, 10 mg.; inositol, 100 mg.; choline chloride, 1 g.; para amino benzoic acid, 10 mg.; nicotinic acid, 20 mg. The reference diet was the same supplemented daily with 200 µg. calcium pantothenate per rat.

Trypanosome counts were made some by the method of Taliaferro (1924) and some by that of Kolmer (1915). Red- and white-cell counts were made by standard haemocytometer methods. Differential white cell counts were made on blood films stained with Wright's. The presence or absence of division forms of *T. lewisi* was determined by inspection of the stained blood films. Uniform size and form are, of course, indicative of adulthood, or the non-multiplicative condition, while variability of size, form, and stainability, as well as concrete evidence of division of cell organelles, indicate reproduction.

#### EXPERIMENTAL DATA

Data composed of weight increases of the hosts and numbers of trypanosomes per cubic millimeter of blood are summarized in Table 1. It is apparent in all six of the experiments, in which the rats of six litters were employed, that the deficient rats failed to make the weight gains registered for the recipients of pantothenate. On the other hand, the trypanosome population in the deficient rats attained heights not equaled in the normal rats. It is true, however, that the trypanosome numbers in individuals of the latter often exceeded certain ones of the former, and that there were individuals which did not seem to exhibit decreased resistance by reason of the decreased dietary deficiency. An example of the latter is Rat No. 3, harboring a population of 2,000 trypanosomes per cubic millimeter of blood on the tenth and twelfth days. It is entirely possible, however, that such a rat, if it had been fed pantothenate, would have reacted either like Rat No. 8, which did not exhibit trypanosomes in the circulating blood, or like Rats Nos. 40 and 42 in which they were seen but not in sufficient numbers to be counted.

An analysis of the trypanosome numbers appearing in Table 1 shows the following means of the maximum populations (in tens of thousands) attained in the nonrecipients of pantothenic acid (A) and the recipients (B):

Experiment 1, (A) 131 and (B) 21; Exp. 2, (A) 102 and (B) 19; Exp. 3, (A) 99 and (B) 15; Exp. 4, (A) 51 and (B) 7; Exp. 5, (A) 32 and (B) 5; Exp. 6, (A) 19 and (B) 2.

Thus the means for the nonrecipients were from 5.4 to 9 times as high as those for the recipients. The record shows a strange decrease in both

sets of means with each succeeding experiment. The true explanation of this phenomenon is not yet known to the writers, but it may be that as the microorganism was passed from rat to rat of our strain it regained that tendency, which was commented upon in the previous report, to appear only in small numbers in the circulating blood.

Microscopic examinations of stained blood smears confirmed that, as in the previous work, the multiplicative phase of the *T. lewisi* infection was abnormally prolonged in the deficient rats. Without exception, the parasite population attained adulthood (defined above) by the eighth, ninth, or tenth day in the pantothenate recipients, whereas there were a number of exceptions to this behavior among the deficient rats, as indicated by flagellates with dividing or divided parabasal bodies and nuclei, double flagella, and variability in body size and shape. The most marked exceptions occurred in Rats 2, 4, 10, 14, 15, and 16, in whose blood evidence of division forms was detected at least as late as the fourteenth or fifteenth days. Rats 14 and 16 presented especially interesting cases, for division forms appeared in them continuously from May 10 (fourth day of the infection) to July 11 (sixty-sixth day). The condition of the rats became so severe by the latter date that, in order to save them, the stock-growing ration was fed. The condition of the rats rapidly improved, as indicated by increasing weight, hair growth, and better care of the coat, but size variability persisted in the flagellate population for two weeks.

Rats 14 and 16 were of special interest for another reason: their parasitemias apparently reached a peak on about the fifteenth day and steadily declined thereafter, although division forms persisted. The day of the infection (parenthesis) and trypanosome numbers in hundreds of thousands per cubic millimeter of blood for rats 14 and 16, respectively, were recorded as follows: (10) 1,060 and 810; (12) 890 and 790; (15) 1,260 and 1,580; (18) 505 and 239; (22) 375 and 191; (24) 351 and 261; (34) 72 and 249; (40) 140 and 130; (66) 50 and 42.

Only two deaths due to trypanosomiasis occurred among the deficient rats, Rat 2 on the fifteenth day of its infection, and Rat 15 on the seventeenth. Division forms were found in the blood of both rats on the day preceding death, and both showed evidences of extreme red cell anemia; namely, pallor of eyes and mucous membranes, and occurrence of normoblasts and reticulocytes in the smears. Counts were not made on Rat 2, but in Rat 15 the red cell count dropped from 6 million on the day of inoculation to 1.2 million on the fifteenth day. It was a curious coincidence that on the latter date the numbers of trypanosomes and red cells per cubic millimeter of blood were practically identical.

It will be noted in Table 1 that the weight increases were in general considerably higher in the infected pantothenate recipients than in the infected deficient rats. Experiment 5 shows that this is by no means entirely due to the vitamin deficiency, for the weight gains up to the seventeenth day were considerably higher in the uninfected deficient rats than in the infected deficient rats.

The development of red cell anemia in the pantothenate series was

found to be a common occurrence. A litter of eleven young rats averaging about 67 grams was selected for special study. Nos. 28-31 and A-D were started on the pantothenate deficient ration, and Nos. 32-34 on the same plus pantothenate. All except A-D were inoculated with *T. lewisi* on the fourteenth day. Red cell counts on the inoculation date and the seventh, tenth, thirteenth, and eighteenth day of the ensuing infection are recorded in Table 2. The trypanosome counts on the seventh, tenth, twelfth, and sixteenth days are recorded in Table 1.

Trypanosome numbers were, as expected, considerably higher in the deficient series than in the normals (Table 1). Likewise, as previous observations had led us to expect, red cell anemia developed in all four infected deficient rats, but not to any considerable degree in the others. In certain other instances we had found anemia commencing as early as the seventh day, and in this series likewise it seemed to commence about that date and to become extreme by the tenth and thirteenth days. The severity of the anemia in these cases was in direct proportion to the parasitemia, an impression we had gained from previous observations. Anemia did not develop in the four uninfected deficient rats.

The differential white cell counts for the deficient series (Rats 28-31) showed an increase in neutrophils at the expense of the lymphocytes on the seventh, tenth, and thirteenth days, with some readjustment toward the normal by the eighteenth day (Table 2). There was a higher eosinophilia in the normal series than in the deficient or uninfected series, but the observations are too few to warrant definite conclusions. The evidence for a basophilia in the deficient series is, however, strong (see Table 2). The increase in total white cell counts was more marked and prompt in the normal than in the deficient series.

### SUMMARY AND CONCLUSIONS

Since our previous work on the effect of pantothenic acid deficiency on *Trypanosoma lewisi* infection was complicated by intercurrent Bartonella infection, another attack on the problem was made with a Bartonella-free line of the microorganism and louse-free rats. In the new work the deficiency was produced by withholding pantothenic acid from the diet rather than by the drastic process of dry-heating previously employed. Also, a complete vitamin supplement was fed, save for the absence of pantothenate in the deficient series:

In each of the six test experiments, employing in all twenty-one young rats in the deficient series and the same number in the normal series, the parasitemia was abnormally exalted in the hosts from whom pantothenate was withheld. Likewise, the multiplicative period of the infection was abnormally prolonged in the deficient hosts whose parasite populations were the most enhanced.

Other aspects of the infection in pantothenate deficient rats were (1) anemia, (2) neutrophilia, (3) basophilia, (4) less than normal and more delayed increase in total white cell count, (5) either reduced growth

TABLE 2

BLOOD CELL CHANGES IN INFECTED NONRECIPIENTS OF PANTOTHENATE (28-31), INFECTED RECIPIENTS (32-34), AND UNINFECTED NONRECIPIENTS (A-D)

(Started on diets June 14 when 40 days of age and infected June 28)

Rat No.	P.A.	Date	Red Cells (1,000,000)	White Cells (1,000)	Lymph. %	Neutr. %	Mono. %	Eos. %	Bas. %
28...	—	6-28	7.2	6.6	72	20	7	1	.....
		7-5	6.8	4.2	60	33	6	.....	1
		7-8	4.2	6.3	59	36	3	1	1
		7-11	4.4	6.8	62	34	2	.....	2
		7-16	5.4	6.8	61	31	6	.....	2
29...	—	6-28	7.2	4.9	83	12	5	.....	.....
		7-5	6.1	4.9	56	40	2	1	1
		7-8	2.4	4.5	54	40	1	1	4
		7-11	1.8	10.7	53	45	.....	.....	2
		7-16	4.3	6.2	60	37	3	.....	.....
30...	—	6-28	9.9	4.7	78	20	2	.....	.....
		7-5	7.7	4.4	62	34	2	.....	2
		7-8	3.5	5.6	60	40	.....	.....	.....
		7-11	2.5	10.4	61	36	1	.....	2
		7-16	3.8	9.0	74	21	4	1	.....
31...	—	6-28	6.2	5.1	74	22	4	.....	.....
		7-5	5.8	4.7	44	51	4	.....	1
		7-8	4.5	4.3	75	23	2	.....	.....
		7-11	3.0	6.9	38	56	3	.....	3
		7-16	2.8	7.8	72	24	4	.....	.....
32...	+	6-28	6.9	5.0	56	42	1	1	.....
		7-5	7.5	8.9	50	41	8	1	.....
		7-8	6.7	11.0	86	12	2	.....	.....
		7-11	5.9	9.0	68	25	5	2	.....
		7-16	5.8	7.6	86	12	2	.....	.....
33...	+	6-28	4.9	7.0	74	22	4	.....	.....
		7-5	6.2	8.9	78	20	2	.....	.....
		7-8	6.1	13.3	88	11	1	.....	.....
		7-11	4.6	9.8	64	28	6	2	.....
		7-16	7.0	13.0	73	21	6	.....	.....
34...	+	6-28	5.6	6.5	69	19	11	1	.....
		7-5	6.9	7.0	74	24	1	1	.....
		7-8	7.0	10.5	75	23	1	1	.....
		7-11	6.9	9.0	66	30	4	.....	.....
		7-16	7.0	8.8	70	26	2	1	1
A....	—	6-28	7.9	6.4	89	9	2	.....	.....
		7-5	7.4	8.4	84	10	6	.....	.....
		7-8	6.2	6.9	85	10	5	.....	.....
		7-11	7.2	5.9	81	14	5	.....	.....
		7-16	7.5	6.0	72	21	5	1	1
B....	—	6-28	6.0	4.8	69	23	8	.....	.....
		7-5	7.2	6.9	68	24	7	.....	1
		7-8	7.4	8.3	78	18	4	.....	.....
		7-11	8.8	7.7	68	26	6	.....	.....
		7-16	8.7	7.8	71	22	7	.....	.....

TABLE 2—Continued

Rat No.	P.A.	Date	Red Cells (1,000,000)	White Cells (1,000)	Lymph. %	Neutr. %	Mono. %	Eos. %	Bas. %
C....	—	6-28	7.6	3.6	76	20	3	1	.....
		7-5	7.5	9.6	79	20	1	.....	.....
		7-8	7.4	8.1	81	15	4	.....	.....
		7-11	7.6	6.0	69	25	5	1	.....
		7-16	8.1	6.5	68	27	5	.....	.....
D....	—	6-28	7.3	5.3	85	12	3	.....	.....
		7-5	6.8	7.0	76	17	6	1	.....
		7-8	6.1	7.2	59	34	7	.....	.....
		7-11	7.5	7.4	60	36	4	.....	.....
		7-16	7.8	7.6	68	28	3	.....	1

rate of host or loss of weight and, (6) in extreme cases, death of the host.

Since anemia, changes in total and differential white cell counts, and sudden adverse effects on growth did not occur in the uninfected deficient controls, it can be said that *T. lewisi* may become a pathogen in pantothenate deficient rats.

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## CURVE FITTING: AN ART OR A SCIENCE?

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Graphical curve fitting is done in accord with the judgment of the fitter: the curve may reflect his judgment more faithfully than it does the fitted points. Often the data are not sufficiently plentiful to afford any objective comparison between curves fitted by eye and by least squares; hence, the recent report of a successful graphical fitting with 216 sets of observations (Becker, Carter, Burks, and Kaleita: this *Journal*, Volume 20, No. 4, July, 1946, pages 403-13) was seized upon as an opportunity to compare methods.

After abandoning a direct fitting of the data to the desired dependent variable, plasma atabrine, A.P., the authors (Becker, *et al.*) discovered a quantity,  $F = A.U. / (T.A. \cdot A.P.)$ , which followed a notable pattern in the plane of the two independent variables, urinary atabrine, A.U., and titratable acidity of urine, T.A. So, they marked off the plane into a series of bands within each of which  $F$  was approximately constant. By means of this chart, the two field measurements, A.U. and T.A., determined a value of  $F_m$  from which the plasma atabrine was estimated as,

$$C.A.P. = \frac{A.U.}{(T.A.) (F_m)}$$

In this article two features will be discussed, the fitting of  $F$  and the original problem of estimating A.P.

### THE FITTING OF $F$

The authors ran into two difficulties in fitting  $F$ : one group of six points lay so close to the T.A.-axis that there was some skepticism as to the validity of the results, and one point was found at the opposite extreme, very close to the axis of A.U. These seven points were, in effect, omitted from the graph. It was partly to learn the sources of these difficulties that the surface now to be described was fitted to the 216 points.

As a first trial, it was deemed adequate to fit the general quadratic function, the result being,

$$F = 108 + 0.028 A.U. - 53T.A. - 0.0000004 (A.U.)^2 + 5.4 (T.A.)^2 - 0.003 (A.U.) (T.A.)$$

Multiple  $R^2$  was only 0.3581. One of the authors' difficulties was still in evidence: the 216th point, lying close to the vertical axis A.U., had the deviation from the fitted surface,  $888 - 176 = 712$ . This single point was responsible for 71 per cent of the entire sum of squares of deviations

from regression. For the objectives in view it seemed wise to omit this point as the authors effectively did. Upon doing this the equation became

$$F = 85 + 0.023 \text{ A.U.} - 37 \text{ T.A.} - 0.0000003 (\text{A.U.})^2 + 3.61 (\text{T.A.})^2 - 0.002 (\text{A.U.}) (\text{T.A.}), \text{ with } R^2 = 0.5941.$$

Some light is now thrown on the other difficulty that the authors mentioned. The calculated values of  $F$  for the first six points are,

$$-8, -6, 1, -6, -3, \text{ and } 10$$

The four negative  $F$ 's lead to negative estimates for A.P. From this, together with many other negative  $F$ 's and large deviations from regression, it was inferred that there is no polynomial function of A.U. and T.A. (with a moderate number of terms) which is suitable for the data in hand; this may account for the hesitancy of the authors to use the points 1-6.

From the successful estimation of A.P. reported in the second part of this article, the present writers guessed that the logarithms of the variables might be more appropriate for fitting the data. The linear regression in logarithms,

$$\log F = 1.421 - 0.8334 (\log \text{ T.A.} + 1) + 0.6117 (\log \text{ A.U.} - 1)$$

raises  $R^2$  to 0.7781. But a significant amount of curvilinearity was evident, chiefly in  $\log \text{ A.U.}$  It turned out that the data in this particular sample were adequately fitted by

$$\log F = 2.114 - 0.8507 (\log \text{ T.A.} + 1) + 0.1344 (\log \text{ A.U.} - 1)^2 \text{ with } R^2 = 0.7930.$$

The authors' 216th point is still aberrant as judged by this new equation, but their first four points are nicely estimated:

	Points						216
	1	2	3	4	5	6	
Observed $F$ . . . . .	6	13	9	11	25	22	888
Estimated $F$ . . . . .	7.0	9.0	7.4	10.6	10.6	9.4	358

Points 5 and 6, while deviating considerably from regression, are by no means as divergent as others among the 209 points fitted. There is no evident reason, therefore, for excluding these six points from the fitting.

It is possible to make some comparisons between the least squares method just described and the graphical fitting presented by Becker and his fellow workers. From their Table 1, the sum of the squares,  $(\log F - \log F_m)^2 = 1.766$ , was calculated, leading to an estimate of their multiple regression,  $R^2 = 0.8578$ , greater than the best reported above, 0.7930. While no exact test of significance is available, a rough approximation may be attempted. Nine of the curves drawn by the authors were parabolic in form, but with some obvious divergencies to follow the

data. Perhaps three or four degrees of freedom may be assigned to them; say 33 for the nine. The 10th irregular closed curve (which apparently didn't greatly improve the fitting) can scarcely have required less than five degrees of freedom, making 38 in all. This leads to the following conjectural analysis of variance applied to 209 points:

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Square
Total deviations of logarithms . . . . .	208	12.421	.....
Constants in least squares regression . . . . .	2	9.850	4.925
Deviations from least squares regression . . . . .	206	2.571	0.0125
Deviations from graphical fitting . . . . .	170	1.766	0.0104
Constants in graphical fitting . . . . .	38	10.655	0.280
Excess of constants, graphical over least squares . . . . .	36	0.805	0.0224

$$F = 0.0224/0.0104 = 2.15 \quad F_{.01} = 1.75$$

If this procedure be reasonable, it becomes clear that the graphical fitting is the more effective in respect of both average deviation from regression and total reduction in sum of squares. But this advantage is gained at the expense of reduction in sum of squares per constant fitted. Here the number of constants used in the graphical fitting exacts no penalty because of the many degrees of freedom available.

#### THE ESTIMATION OF A.P.

The ultimate objective of Becker and his collaborators was to estimate the atabrine concentration in the blood plasma by means of simple field measurements of urine characteristics. The most obvious relation to try was the correlation between the concentrations of atabrine in the plasma and in the urine. The report was that, "when plasma levels (A.P.) were plotted on the ordinates and corresponding urinary levels (A.U.) on the abscissas in the manner of a correlation chart, the widely scattered points demonstrated the lack of any considerable degree of correlation." This finding is not surprising since the correlation is only 0.5, the regression curved, and the variance heterogeneous. Since ratios were used effectively throughout the authors' investigation, it seemed worth while to the present writers to examine the correlation between the logarithms of A.P. and A.U. These logarithms were found to follow the normal bivariate distribution fairly closely with  $r^2 = 0.5682$ . This is comparable with the authors' final correlation between computed and observed plasma atabrine levels,  $R^2 = 0.5722$ . Had these investigators been fortunate enough to have observed this relation among the logarithms, they could have attained their objective by the use of the simple graphical computing device illustrated in Figure 1, where field determinations of urinary atabrine (A.U.) may be located on the upper scale and corresponding estimates of plasma atabrine (A.P.) read on the lower. The calculation

of C.A.P. would have been avoided, and the resulting estimates of A.P. would have been practically as accurate, on the average, as those designated as C.A.P.

Other investigators had used a relation that suggested

$$A.P. = k A.U./T.A.$$

The authors tried plotting the ratio, A.U./T.A., against A.P. but were disappointed. It turns out that the logarithms are linearly related,

$$\log A.P. = 0.429 \log A.U. - 0.212 \log T.A. - 0.104,$$

with  $R^2 = 0.6393$ . From internal evidence, this would seem to be the best fitting equation that can be found: there is no regular (curved) deviation

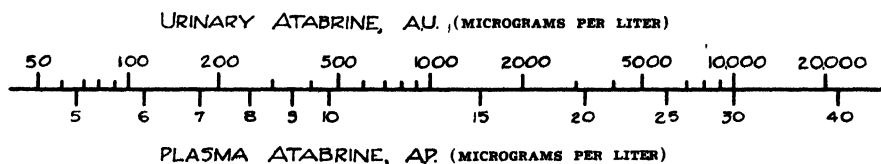


FIG. 1.—Scales for estimating Plasma Atabrine (A.P.) from Urinary Atabrine (A.U.). The equation is  $\log A.P. = 0.3595 \log A.U. + 0.0398$ .

from it, and the variance of the deviations is reasonably homogeneous. Use of this equation might be forbidding to those not accustomed to logarithms, especially if they are in the field, but the graphical solution in Figure 2 is easier than the combination of chart reference with computation which was furnished by the authors. The graphical calculation illustrated is for case No. 5, which the authors placed in Group A<sub>1</sub> without attempting to estimate the plasma atabrine. The deviation from regression for this case is  $4 - 8 = -4$ , substantially less than the median deviation which is about 6.5. In fact, none of the members of this group show as much as average fluctuation from the regression.

Retransformed to the original units, the equation,

$$A.P. = 0.787 \frac{A.U.^{0.429}}{T.A.^{0.212}},$$

indicates why the authors failed to find a linear relation between A.P. and A.U./T.A. The two independent variables are not equally effective in estimating A.P., the standard partial regression coefficients of  $\log A.P.$  on  $\log A.U.$  and  $\log T.A.$  being, respectively, 0.900 and  $-0.304$ .

It is interesting to reconsider case No. 216 which was treated as aberrant because of its large deviation from estimated F. Its  $A.U. = 3,220$  and  $T.A. = 0.3$  applied to Figure 2 give an estimated value of  $A.P. = 33$  as compared to the observed value, 12. The deviation,  $-21$ , is large; but it is exceeded in magnitude by the deviation for case No. 164, which is 28, and is not much greater than the deviation for case No. 192, which is

18. Thus, if the authors had not been diverted by their discovery of F, they would probably not have rejected any of their 216 sets of observations.

The worth of any empirical curve fitting must be judged ultimately by success in predicting behavior in other samples from the same population. In an addendum the authors note the fact that a hundred additional determinations had been available for study. These would have comprised a valuable means for testing the results which we have described, but Dr. Becker tells us that they were war casualties.

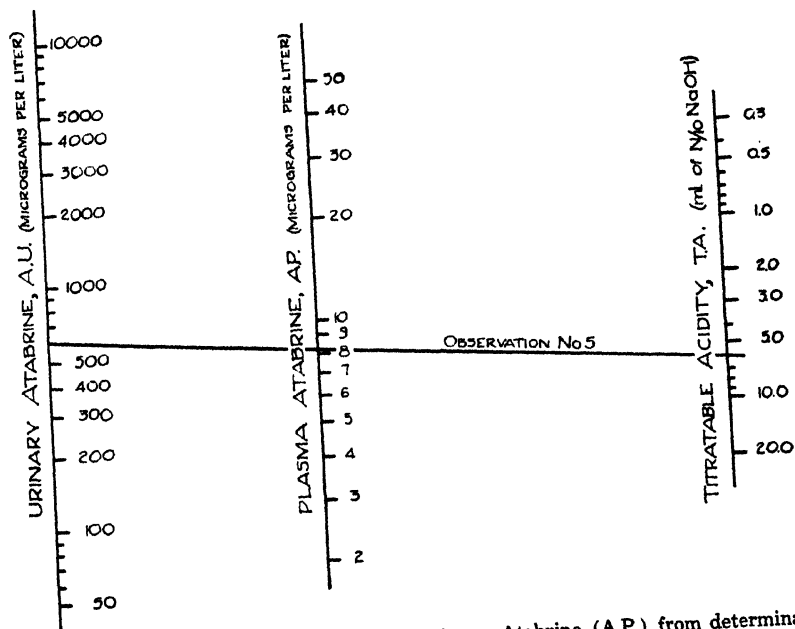


FIG. 2.—Alignment chart for estimating Plasma Atabrine (A.P.) from determinations of Urinary Atabrine (A.U.) and Titratable Acidity (T.A.) of the urine. The oblique line illustrates the estimation of A.P. = 8 from the observed values for case No. 5; A.U. = 600, T.A. = 6.

The answer to the question of the title seems to be that curve fitting is, in large measure, an art; but a modicum of science may not be amiss.

#### SUMMARY

Becker, Carter, Burks, and Kaleita were successful in fitting the function F to the data described in the July, 1946, number of this *Journal*. The evidence is that no continuous surface with a moderate number of degrees of freedom would fit the data so closely as did their graphical curves. There is some indication, however, that the population regression

may be almost as well described by a simple logarithmic function as by their graph.

As compared with the authors' method, the estimation of plasma atabrine (A.P.) can be done as effectively and more conveniently by the use of either of two graphical computing devices which are presented in Figures 1 and 2.

# OXIDATION OF POLYHYDRIC ALCOHOLS BY *ACETOBACTER SUBOXYDANS*<sup>1</sup>

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From a physicochemical viewpoint, fermentation is a special case of catalysis, or rather autocatalysis, in heterogeneous system. The catalysts, the enzymes, are manufactured during the course of the reaction through the growth of the microorganism involved. Deliberate use of microorganisms as a series of catalysts, of graded oxidizing and reducing tendencies, may be illustrated by the oxidation of polyhydric alcohols by species of the genus *Acetobacter*. The reaction is one of dehydrogenation of a secondary alcohol group and thus furnishes a simple model from the viewpoint of physical chemistry.

As early as 1852, Pelouze (27) subjected the juice of the mountain ash berry to spontaneous fermentation in open vessels. After fourteen months, he obtained from the fermented liquors a crystalline material with the molecular formula  $C_6H_{12}O_6$ ; the compound was levorotatory and reduced Fehling's solution. In 1896, Bertrand (1) proved this compound, sorbose, to be formed by bacterial action upon the polyhydric alcohol sorbitol. This "sorbose bacterium" is now known as *Acetobacter xylinum*. Bertrand (1,2,3,4,5,6,7,8) reported the oxidation of eight different sugar alcohols by the organism to give the corresponding ketose sugars. The oxidative action proved to be highly specific. Only the secondary alcohol group next to the primary alcohol group on one end of these polyhydric alcohols was found to be oxidized. Moreover, Bertrand (2) stated that the contiguous secondary alcohol group must be in the *cis* position. He foresaw many uses of this reaction in carbohydrate chemistry.

Subsequently it was found that several species of the genus *Acetobacter* promote similar oxidations. An excellent survey of the biochemical activities of the acetic acid bacteria has been given by Butlin (10). Kluyver and DeLeeuw (25) isolated *Acetobacter suboxydans*, which has been most widely used in recent years due to its superior cultural characteristics and oxidative behavior. As will be shown later, it is even more specific in its reactions than is *Acetobacter xylinum*.

In our laboratories, the main emphasis has been placed on developing conditions for obtaining maximum yields of the keto-compounds and methods for their isolation and purification. The first part of this paper deals with this phase of the problem, to be followed by a general dis-

<sup>1</sup> Presented before the Division of Sugar Chemistry and Technology at the 110th Meeting of the American Chemical Society, Chicago, Sept. 11, 1946.



cussion of stereochemical considerations of special interest to carbohydrate chemists.

#### SORBITOL TO SORBOSE

In 1935 a fellowship was established in our laboratories, by two pharmaceutical concerns to explore the possibility of the microbial synthesis of ascorbic acid or Vitamin C. The proposed steps involved the oxidation of sorbitol to sorbose and the conversion of the latter to ascorbic acid. The present discussion deals only with the conversion of sorbitol to sorbose. Bertrand and others had prepared sorbose by the action of *Acetobacter xylinum* on media containing only 3 per cent to 6 per cent of sorbitol. The yields of sorbose were relatively low and fermentation periods of several weeks were required.

Four species of the genus *Acetobacter* were tested in our laboratories on a medium containing 0.5 per cent yeast extract (Difco) and 15 per cent sorbitol, a much higher concentration of substrate than had been previously employed. The relative yields of sorbose are shown in Table 1. This relative order proved identical to that given by Kluyver and De-

TABLE 1  
RELATIVE YIELDS OF SORBOSE BY FOUR SPECIES OF THE GENUS *Acetobacter*

Organism	Relative Yield of Sorbose
<i>Acetobacter suboxydans</i> .....	100
<i>Acetobacter aceti</i> .....	85
<i>Acetobacter xylinum</i> .....	75
<i>Acetobacter peroxydans</i> .....	5

Leeuw (25) for the increasing oxidizing tendency of these species of *Acetobacter*. The *Acetobacter peroxydans* proved so active as to give relatively large quantities of carbon dioxide and water, and little sorbose. It is evident that *Acetobacter suboxydans* is the best organism for the reaction under consideration and it was employed in all of our studies on the oxidation of polyhydric alcohols. This organism has a further advantage over *A. aceti* and *A. xylinum* in that it does not grow in a sticky, cellulosic mat, but forms a friable growth permitting easy removal and separation from the fermented medium.

Preliminary experiments with *A. suboxydans* in the oxidation of sorbitol showed the optimum temperature to be from 25° to 30° C.; pH from 5.1 to 6.8; and that at least 0.5 per cent of yeast extract must be present. Later it was found that these conditions also held for the oxidation of the other polyhydric alcohols studied. Hence, the following conditions were maintained in all subsequent work: 28° C., pH = 6.1, and 0.5 per cent yeast extract (Difco). The pH of 6.1 was most convenient since all media were at or near this value when made up and sterilized.

It was found that disturbing the flasks to take samples materially reduced the yields of oxidation product. Hence, each datum presented

represents the analysis of a separate flask. Proper aeration of the culture speeds up the rate of oxidation and obviates the necessity of care in not disturbing the surface growth. However, difficulties in obtaining uniformity of aeration and duplication of equipment precluded its use in running series on a large number of small flasks.

Using a medium containing 10 per cent sorbitol, data were obtained on the effect of the surface-volume ratio on the yields of sorbose. Typical data are shown in Table 2. It is evident that both the reaction rate and

TABLE 2  
EFFECT OF SURFACE-VOLUME RATIO UPON THE PER CENT YIELD OF SORBOSE  
BY THE ACTION OF *Acetobacter suboxydans*  
(10 Per Cent Sorbitol Medium)

Days	Surface-Volume Ratio (cm. <sup>2</sup> per cc. medium)					
	2.360	1.195	0.589	0.345	0.200	0.119
1.5.....	78	67	41	28	15	10
2.5.....	84	82	58	42	26	14
3.5.....	84	84	77	56	35	21
4.5.....	85	84	80	63	39	24
5.5.....	87	84	81	72	45	30
6.5.....	87	84	82	80	48	.....
7.5.....	89	84	82	80	53	31

final yield increase with increase in surface-volume ratio. Since similar relations held for the other polyhydric alcohols studied, a surface-volume ratio of about 1.195 was used in all subsequent experiments.

In Table 3 are given typical data showing the effect of the concentration of sorbitol on the yield of sorbose. It is evident that while the rate of oxidation decreases with increasing concentration of substrate, the final yield of sorbose is practically independent of the sorbitol concentration up to and including 35 per cent; there was a marked drop in final

TABLE 3  
EFFECT OF CONCENTRATION OF SORBITOL UPON THE PER CENT YIELD  
OF SORBOSE BY *Acetobacter suboxydans*

Percentage Sorbitol	Days				
	2	3	7	11	14
10.....	80	83	84	.....	.....
15.....	72	.....	86	85	84
20.....	.....	64	80	83	83
25.....	.....	49	73	82	85
30.....	.....	34	62	76	78
35.....	16	.....	67	81	80
40.....	3	.....	9	14	14
45.....	2	.....	3	4	4
50.....	2	.....	.....	2	2

yield at 40 per cent. With 35 per cent sorbitol the concentration of sorbose reached the high value of 28 grams per 100 ml. of fermented medium.

The employment of these high concentrations of sorbitol affords an easy method for the large scale production of sorbose with the handling of minimum volumes of liquids. The sorbose is readily recovered by filtering the fermented medium and evaporating it to the required volume for crystallization. Details of the initial experimental work were published by Fulmer, Dunning, Guymon, and Underkoffler (17).

The rapid development of the process may be shown by the following sequence. At the time of initiation of the project one chemical house offered five grams of sorbose "price quoted on request." Within three months we were preparing sorbose in 15 pound lots, production being limited only by requirements and equipment. Colleagues in the Agricultural By-Products Laboratory, at Ames, studied the fermentation in their special rotating drum, developed for the production of gluconic acid by submerged growth of *Aspergillus niger*, under aeration. Soon they were preparing sorbose in 200 pound lots. Their results on the pilot-plant scale production of sorbose were published by Wells, Stubbs, Lockwood, and Roe (41) just two years following the initiation of our project. Within a short time this fermentation formed the first step in the industrial production of synthetic ascorbic acid.

#### MANNITOL TO LEVULOSE

In the conversion of mannitol to levulose by *Acetobacter suboxydans* it was found by Fulmer, Dunning, and Underkoffler (18) that while the rate of oxidation decreased with increasing concentration of substrate, the final yield was practically independent of the concentration of mannitol up to and including 25 per cent; there was a marked decrease in final yield at 30 per cent. With 25 per cent mannitol the yield of levulose was better than 90 per cent of theory after seven days. Evidently both sorbitol and mannitol are efficiently oxidized at relatively high concentrations.

#### GLYCEROL TO DIHYDROXYACETONE

In the conversion of glycerol to dihydroxyacetone the concentration of the substrate should not exceed 6 per cent for maximum yields with surface cultures. This is in contrast to sorbitol and mannitol which are efficiently oxidized at concentrations of at least 25 per cent. Incubation beyond seven days did not give increased yields, at which period the yield of dihydroxyacetone was 90 per cent of theory or better. Methods were developed for the isolation of the dihydroxyacetone by which 80 per cent of the product was obtained in the crystalline form. The details were published by Underkoffler and Fulmer (36).

This work made dihydroxyacetone, a highly important ketotriose, available to American chemists and biochemists. Subsequent to the publication mentioned, experience has led to modifications which simplify the preparation, recovery, and purification of the dihydroxyacetone.

The medium employed contains 5 g. of pure glycerol, 0.5 g. of Difco

yeast extract, and 0.25 g. of monopotassium phosphate per 100 ml. It is distributed in flasks which are plugged with cotton and sterilized by heating at 15 pounds steam pressure for 15 to 30 minutes. For surface culturing 300 ml. of medium are used in each 2-liter Erlenmeyer flask. For submerged culturing any convenient size of flask may be employed which is about two-thirds filled with the medium and equipped for aeration with sterile air through efficient air dispersers such as those made of alundum. The sterile medium is inoculated with 2 to 5 per cent of a 24-hour culture of *Acetobacter suboxydans* grown on a medium of the same composition and then incubated at 28° C. Surface cultures are incubated for seven to ten days without disturbing the flasks and submerged cultures are incubated for three to seven days with continuous aeration at the rate of at least 200 ml. of air per liter per minute. If desired the completion of the fermentation may be determined by periodic reducing sugar analyses, the incubation being interrupted when the maximum reducing value has been reached.

The fermented liquid is worked up in the manner described by Underkoffler and Fulmer (36) to the point of crystallizing the sirup. The sirup is placed in a beaker over sulfuric acid in a vacuum desiccator which is evacuated with a Hyvac pump until the mixture boils. The vacuum is renewed at least twice each day for two or three days, each time to the point where there is an actual boiling of the sirup. After this period the vacuum is released and the thick sirup seeded with a few crystals of dihydroxyacetone and the vacuum again applied. In two or three days after seeding, the mass crystallizes to a solid, glassy consistency. The beaker is placed in an ice bath and a half-volume of cold absolute alcohol added. The mass is worked with a spatula until it can be transferred to a cold, dry mortar and is there triturated to a smooth paste. The material is filtered by suction, the solid again triturated with a small amount of cold absolute alcohol and again filtered by suction. The product is thoroughly dried over calcium chloride in a vacuum desiccator. The crude dihydroxyacetone so obtained is slightly colored and is stored in a tightly stoppered dark glass bottle in the refrigerator, being further purified for use as required. Additional quantities of the compound may be obtained from the mother liquor and wash liquid by working up in the same manner as before.

The crude dihydroxyacetone is recrystallized from hot absolute alcohol, taking care to prevent access of moisture since traces of water prevent satisfactory crystallization. The crude material is dissolved in boiling absolute alcohol. If the solution is colored, it is decolorized (while hot) with Norite and filtered by suction. Most of the alcohol is distilled off by using a wire gauze and open flame under the flask. The residual small volume of material is cooled thoroughly in a well-stoppered flask, seeded with a few crystals of pure dihydroxyacetone and shaken. After crystallization has started, the stoppered flask is placed in the refrigerator for a few hours, with occasional shaking. The solid is filtered on a cold filter by suction and washed with cold absolute alcohol until it is pure

white. It is then dried over calcium chloride in a vacuum desiccator. Additional amounts of the pure crystalline dihydroxyacetone can usually be obtained by distilling the alcohol from the combined mother liquors and wash liquids and following the same procedure as before. The pure dihydroxyacetone should be stored in a well-stoppered dark glass bottle in a cool place, preferably in a refrigerator.

The pure dihydroxyacetone prepared as above is the usual bimolecular alpha-modification. The crystals are colorless, prismatic plates, very soluble in water, sparingly soluble in organic solvents, and melt indefinitely at 68°–80° C. The unimolecular beta-modification may be obtained by the method of Fischer and Mildbrand (15) by distilling the dihydroxyacetone under high vacuum.

It is preferable to purify and recrystallize the dihydroxyacetone shortly before use, since on standing it spontaneously condenses through loss of water into a pasty mass containing substances of higher molecular weight and of limited solubility in water. This change occurs much more slowly when the purified material is stored in the dark in the refrigerator.

The best method of purifying dihydroxyacetone which has undergone this decomposition appears to be that of Reeves and Renbom (31) as follows: Suspend 50 g. of the pasty material in 100 ml. of pure acetone and shake continuously for 24 hours at room temperature (best below 25° C.). Filter with suction, wash with acetone and dry thoroughly over calcium chloride in a vacuum desiccator. After the acetone treatment and washing, the material may be recrystallized immediately from absolute alcohol as described above.

#### ERYTHRITOL TO ERYTHRULOSE

Whistler and Underkoffer (42) studied the oxidation of *meso*-erythritol to L-erythrulose and found the concentration of substrate should not exceed 4.5 per cent. Under optimum conditions the yield of L-erythrulose was practically quantitative in seven days. Methods were developed by which 87 per cent of the erythrulose was recovered from the fermented medium as a colorless sirup.

#### OXIDATION OF *meso*-INOSITOL

Studies were made on the oxidation of the cyclic polyhydric alcohol *meso*-inositol (or *i*-inositol) and reported by Dunning, Fulmer, Guymon, and Underkoffer (13) and Dunning, Fulmer, and Underkoffer (14). Since this compound contains only secondary alcohol groups it offered an interesting test of the relation of configuration to the action of the organism. Moreover, this type of oxidation, if successful, would permit the preparation of cyclic ketones not heretofore available. *meso*-Inositol is also of biological interest because of its wide distribution in nature as a component of phytin, its identity with the yeast growth stimulant Bios I and the fact that it is also classed as a vitamin.

The development of the medium for the oxidation of inositol furnished an especially interesting problem. The culture could not be carried be-

yond the fifth transfer on an inositol-yeast extract medium. However, the presence of as little as 0.025 to 0.05 per cent of sorbitol permitted continuous subculture and a high conversion of the inositol to a reducing compound. At first it was thought that some factor was formed by the action of the organism on sorbitol favoring the growth and oxidative action on the inositol. Further studies, however, showed that the sorbitol merely served as a substrate allowing the growth of the organism with accompanying production of enzymes capable of oxidizing the inositol. That is, *A. suboxydans* can assimilate and oxidize sorbitol; it cannot assimilate but can oxidize the *meso*-inositol. Low concentrations of other assimilable substances such as glycerol, erythritol, dextrose, and mannitol were found to serve the same purpose as did sorbitol. However, the presence of such assimilable substrates did not permit the oxidation of dulcitol or of L-rhamnitol. compounds, not attacked by the organism because of their unfavorable configurations.

Since methods for preparation, recovery, and purification of the oxidation product of *meso*-inositol have not been previously published, they are now presented.

The medium employed contains 3 g. of *meso*-inositol, 0.05 g. of sorbitol, and 0.5 g. of Difco yeast extract per 100 ml. It is distributed in flasks, sterilized, inoculated, and fermented in the same manner as described above for the preparation of dihydroxyacetone. Upon completion of the fermentation a sufficient amount of lead acetate solution is added to give a final concentration of about 0.75 per cent. Then 10 g. each of Norite and diatomaceous earth per liter are added, the mixture is thoroughly shaken and filtered by suction. Excess lead is removed from the filtrate by treating with hydrogen sulfide and the solution filtered with suction. The clear filtrate is concentrated by vacuum distillation to about one-half the original volume, and again treated with hydrogen sulfide. After suction filtration the evaporation is continued *in vacuo* until crystallization begins. The material is then placed in the refrigerator overnight for completion of precipitation, the solid finally being filtered off and washed with 50 per cent alcohol. The yield is as much as 75 g. of product per 100 g. of *meso*-inositol originally present in the fermentation medium.

The crude product is only slightly soluble in cold water but dissolves in boiling water to give a brown colored solution. Norite is added, the mixture boiled, and then filtered to obtain a clear, colorless solution. The solution is evaporated *in vacuo* to the point of crystallization, then alcohol is stirred in until a concentration of 60 to 70 per cent alcohol is reached. The mixture is allowed to remain in the refrigerator overnight to ensure complete crystallization and is then filtered. Purification consists in recrystallizing at least once more from water by the above procedure and then two or three times by precipitation with alcohol. The latter is accomplished by dissolving the solid in a minimum quantity of boiling water, and then slowly adding alcohol while the mixture is maintained at the boiling temperature until a concentration of 60 to 70 per cent alcohol is reached. The solution is then cooled and the solid recovered by suction filtration.

The physical properties of the compound depend upon the method employed for the final crystallization. When crystallized by precipitation with alcohol from the boiling aqueous solution, a macro-crystalline product is obtained which is soluble in water. When crystallized from water by evaporation and cooling, a micro-crystalline product is obtained which is difficultly soluble in water. The melting point of a given preparation is quite sharp, but may be varied from  $184^{\circ}$  to  $195^{\circ}$  by altering the method of crystallization.

Regardless of the method of crystallization the compound was found to be soluble in warm, dilute alkali, and insoluble in dilute acids, concentrated sulfuric acid, and organic solvents. The physical and chemical characteristics of the compound showed it to be a keto-inositol. To assist in characterizing the compound the amorphous phenylhydrazone and dinitrophenylhydrazone, and the crystalline acetyl derivative were prepared and carefully purified. In Table 4 are given the melting points and analytical data on these derivatives, the values being compared with those given by Posternak (28,29) for two monoketo-*meso*-inositols or "inososes." The first of these, designated as *epi*-inosose, he prepared by chemical oxidation of *meso*-inositol, and the second, designated as *scyllo-meso*-inosose, by oxidation of *meso*-inositol with *A. suboxydans*. Kluyver and Boezaardt (24) had previously reported the isolation of the latter compound upon oxidizing *meso*-inositol with this organism, and believed it to be identical with the inosose previously prepared by Posternak by chemical oxidation. Posternak (29) found the properties of the derivatives of the two inososes to be different. Posternak (30) showed the configuration of the inosose obtained by oxidizing *meso*-inositol with *A. suboxydans* to be related to scyllitol (I) as well as to *meso*-inositol (II),

TABLE 4  
COMPARISON OF INOSOSES WITH THE FERMENTATION KETO-INOSITOL  
OF DUNNING, FULMER, AND UNDERKOFER

Posternak's (28) chemical *epi*-inosose:

Recrystallized compound, M.P.  $198-200^{\circ}$  C.

Phenylhydrazone, M.P.  $220-222^{\circ}$  C.

2,4-Dinitrophenylhydrazone, M.P.  $270^{\circ}$  C.; nitrogen calculated for monoketo-inositol 12.2 per cent.

Acetyl derivative (penta-acetate), M.P.  $106-108^{\circ}$  C.

Biochemical *scyllo-meso*-inosose (24, 29):

Recrystallized compound, M.P.  $200-202^{\circ}$  C.

Phenylhydrazone, M.P.  $184^{\circ}$  C.

Acetyl derivative (penta-acetate), 2 crystalline forms, M.P.  $211^{\circ}$ ;  $147^{\circ}$  C.

Iodine absorbed in alkaline medium by Willstätter-Schudel (43) procedure, 1.87 equivalents.

Keto-inositol of Dunning, Fulmer, and Underkoffer:

Recrystallized compound, M.P.  $184-195^{\circ}$  C.

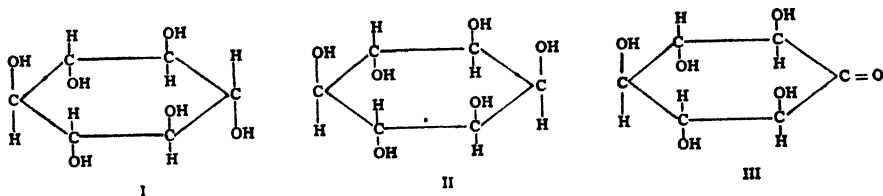
Phenylhydrazone, M.P.  $218^{\circ}$  C.

2,4-Dinitrophenylhydrazone, M.P.  $193-195^{\circ}$  C.; nitrogen calculated for diketo-inositol 20.9 per cent, found 21.0 per cent.

Acetyl derivative (tetra-acetate), M.P.  $114^{\circ}$  C.; acetyl calculated for tetra-acetate of diketo-inositol 50.0 per cent, found 50.4 per cent.

Iodine absorbed in alkaline medium by Willstätter-Schudel (43) procedure, 3.85 equivalents.

which fixed the position of the carbonyl group in the compound, giving the configurational formula (III) for the scyllo-*meso*-inosose.



That is, under the conditions used by Posternak, *A. suboxydans* preferentially oxidizes the middle one of the three *cis* hydroxyl groups of *meso*-inositol.

The data of Table 4 indicate clearly that the product obtained by oxidizing *meso*-inositol with *A. suboxydans* during our investigations was not identical with either of the inososes studied in detail by Posternak. The fact that our product was quite different from the compound later secured by Kluver and Boezaardt (24) and by Posternak (29) with the same organism is difficult to explain. Private communications from workers in several other laboratories in this country have indicated that on oxidizing *meso*-inositol with *A. suboxydans*, certain of these have obtained products resembling in properties the compound studied by Posternak (29), whereas others have obtained products with properties corresponding to the compound obtained in our laboratories. Differences in the strain of *A. suboxydans* employed, cultural conditions, or even conceivably in the source of the inositol, may influence the course of the reaction. Further work will be required to decide this question.

The data of Table 4 indicate our compound to be a diketo-*meso*-inositol. The complexity of further identification is indicated by the fact that fourteen diketo-*meso*-inositols are possible.

Recently Chargaff and Magasanik (11) published a note on the oxidation of stereoisomers of the inositol group by *A. suboxydans*. Through isolation as the phenylosazones, these workers demonstrated the formation of  $\alpha$ -diketo derivatives of inositol by the action of the organism on *l*-inositol and *d*-inositol, and through isolation as the phenylhydrazones, the formation of a monoketo derivative of inositol from *epi*-inositol.

Comparative studies were made on the effects of *meso*-inositol and of our keto-inositol on the growth of yeast. It was necessary to exclude the possibility that the compound might be contaminated with yeast extract which would furnish yeast growth factors. Therefore, the phenylhydrazone derivative was prepared in dilute acid solution and the product recrystallized. The keto-compound was recovered by hydrolysis with benzaldehyde and a little benzoic acid according to the method of Posternak (28,29). The purified compound was found capable of stimulating yeast growth, alone and in a variety of combinations with nine known stimulants, with three types of *Saccharomyces cerevisiae*. The keto-inositol served essentially the same role as *meso*-inositol but in the



majority of cases showed a greater initial growth stimulation. It is interesting to speculate as to the possibility that the interchangeability of the two compounds means that they form a reversible oxidation-reduction system.

### 2,3-BUTANEDIOL TO ACETYLMETHYLCARBINOL

Only recently have the three stereoisomeric forms of 2,3-butanediol become available by other than laborious synthetic methods. As will be seen below, means have now been found for their preparation and isolation by biological means.

In our laboratories methods were developed by Fulmer, Christensen, and Kendall (16) giving nearly theoretical yields of 2,3-butanediol by the action of *Aerobacter aerogenes* on sucrose. The glycol so produced is

slightly dextro-rotatory  $[\alpha]_D^{30} = +1.0$ . This glycol will be referred to subsequently as "Aerobacter glycol." Freezing point data indicated the glycol to consist of about 90 per cent of the *meso*-form.

On the other hand, Ward, Pettijohn, Lockwood, and Coghill (40) reported that *Aerobacillus polymyxa* ferments starchy materials with the production of levo-rotatory 2,3-butanediol,  $[\alpha]_D^{30} = -13.0$ . During the war period both types of fermentation were carried through the pilot plant scale in a number of different laboratories.

Fulmer, Underkoffler, and Bantz (19) found that the "Aerobacter glycol," when acted upon by *A. suboxydans*, gave a yield of 90 per cent of acetylmethylcarbinol. As was the case with *meso*-inositol, the culture could not be carried beyond the fifth transfer on a glycol-yeast extract medium. Similarly, the addition of a very low concentration of an assimilable substrate permitted continuous subculture and rapid oxidation of the glycol. The residual glycol, recovered from the fermented

medium, was dextro-rotatory  $[\alpha]_D^{30} = +10.15$ . Hence, the organism attacked the *meso*-2,3-butanediol but did not oxidize the dextro-rotatory form. This high specificity furnishes a method for separating the two forms. The *meso*-form can best be isolated from "Aerobacter glycol" by fractional crystallization from isopropyl ether by the method of Wilson and Lucas (44).

Underkoffler, Fulmer, Bantz, and Kooi (37) found that the levo-rotatory 2,3-butanediol was oxidized almost quantitatively to acetylmethylcarbinol by *A. suboxydans*. That is, the organism oxidizes the *meso*- and levo-rotatory glycols but does not attack the dextro-rotatory form. By appropriate use of *Aerobacter aerogenes*, *Aerobacillus polymyxa*, and *Acetobacter suboxydans* it is feasible to prepare and isolate the three stereoisomeric 2,3-butanediols.

### THE FOUR GROUPS OF POLYHYDRIC ALCOHOLS

The polyhydric alcohols studied in our laboratories fall into four groups with reference to cultural conditions for maximum yields of oxidation products with *Acetobacter suboxydans*. These groups are:

- I. Oxidized at high concentrations (25 per cent or above)—sorbitol and D-mannitol.
- II. Oxidized at relatively low concentrations—glycerol and meso-erythritol.
- III. Require additional assimilable substrate for continuous sub-culture—meso-inositol, meso-2,3-butanediol and D-(—)-2,3-butanediol.
- IV. Not oxidized even in the presence of an additional assimilable substrate—dulcitol, L-rhamnitol and L-(+)-2,3-butanediol.

### STEREOCHEMICAL CONSIDERATIONS

The discussion of the stereochemistry of the polyhydric alcohols as related to their oxidation by members of the genus *Acetobacter* may well be considered under three categories: (a) oxidation of the sugar alcohols having more than four carbon atoms; (b) oxidation of erythritol and the glycols; and (c) oxidation of the desoxy sugar alcohols.

### OXIDATION OF THE SUGAR ALCOHOLS

Bertrand's work (2,6) led him to the conclusions that *Acetobacter xylinum* oxidizes only those sugar alcohols having the *cis* arrangement of the two secondary alcohol groups adjacent to a primary alcohol group and that only the secondary alcohol group contiguous to the primary alcohol group is oxidized. Subsequent work in several laboratories, employing either *A. xylinum* or *A. suboxydans*, has shown no exception to these rules for sugar alcohols having more than four carbon atoms with a hydroxyl group on each carbon atom.

Data are shown in Table 5 for the oxidation of the pentitols. In this and subsequent tables, *A. suboxydans*, *A. xylinum*, and *A. aceti* are designated as *s*, *x*, and *a*, respectively. A question mark indicates that the alcohol has not been tested. Literature references are given after the designation of organism used. Note in Table 5 that, in accordance with

TABLE 5  
OXIDATION OF PENTITOLS

$\begin{array}{c} \text{CH}_2\text{OH}-\begin{array}{c} \text{H} \quad \text{OH} \quad \text{OH} \\   \quad   \quad   \\ \text{C}-\text{C}-\text{C} \\   \quad   \quad   \\ \text{OH} \quad \text{H} \quad \text{H} \end{array}-\text{CH}_2\text{OH} \\ \text{D-Arabitol} \end{array}$	$s(22)$	$\begin{array}{c} \text{CH}_2\text{OH}-\begin{array}{c} \text{H} \quad \text{OH} \\   \quad   \\ \text{C}-\text{C} \\   \quad   \\ \text{OH} \quad \text{H} \end{array}-\text{CO}-\text{CH}_2\text{OH} \\ \text{D-Xylulose} \end{array}$
$\begin{array}{c} \text{CH}_2\text{OH}-\begin{array}{c} \text{OH} \quad \text{OH} \quad \text{OH} \\   \quad   \quad   \\ \text{C}-\text{C}-\text{C} \\   \quad   \quad   \\ \text{H} \quad \text{H} \quad \text{H} \end{array}-\text{CH}_2\text{OH} \\ \text{Adonitol (meso)} \end{array}$	$s(32)$	$\begin{array}{c} \text{CH}_2\text{OH}-\begin{array}{c} \text{OH} \quad \text{OH} \\   \quad   \\ \text{C}-\text{C} \\   \quad   \\ \text{H} \quad \text{H} \end{array}-\text{CO}-\text{CH}_2\text{OH} \\ \text{L-Adonulose} \end{array}$
$\begin{array}{c} \text{CH}_2\text{OH}-\begin{array}{c} \text{H} \quad \text{OH} \quad \text{H} \\   \quad   \quad   \\ \text{C}-\text{C}-\text{C} \\   \quad   \quad   \\ \text{OH} \quad \text{H} \quad \text{OH} \end{array}-\text{CH}_2\text{OH} \\ \text{Xylitol (meso)} \end{array}$	$s(22), x(2,6)$	No oxidation

Bertrand's rule, the two pentitols with the *cis* arrangement of secondary alcohol groups are oxidized. Xylitol, which does not have this configuration is not attacked by the organism. D-Xylulose and L-adonulose have been isolated from the fermented media and conclusively identified.

Table 6 presents data on the oxidation of the hexitols. Only the first five listed have actually been tested and the results conform to Bertrand's rule. The D-fructose, L-sorbose, and L-allulose have been isolated and conclusively identified. Although D-talitol has not been tested, the rule permits the prediction that it should be readily oxidized to D-tagatose.

TABLE 6  
OXIDATION OF HEXITOLS

$\begin{array}{ccccccc} & \text{H} & \text{H} & \text{OH} & \text{OH} & & \\ &   &   &   &   & & \\ \text{CH}_2\text{OH} & -\text{C} & -\text{C} & -\text{C} & -\text{C} & -\text{CH}_2\text{OH} & \\ &   &   &   &   & & \\ & \text{OH} & \text{OH} & \text{H} & \text{H} & & \end{array}$ <p>D-Mannitol</p>	$\xrightarrow{s(18), x(2,6)}$	$\begin{array}{ccccccc} & \text{H} & \text{H} & \text{OH} & & & \\ &   &   &   & & & \\ \text{CH}_2\text{OH} & -\text{C} & -\text{C} & -\text{C} & -\text{CO} & -\text{CH}_2\text{OH} & \\ &   &   &   & & & \\ & \text{OH} & \text{OH} & \text{H} & & & \end{array}$ <p>D-Fructose</p>
$\begin{array}{ccccccc} & \text{OH} & \text{H} & \text{OH} & \text{OH} & & \\ &   &   &   &   & & \\ \text{CH}_2\text{OH} & -\text{C} & -\text{C} & -\text{C} & -\text{C} & -\text{CH}_2\text{OH} & \\ &   &   &   &   & & \\ & \text{H} & \text{OH} & \text{H} & \text{H} & & \end{array}$ <p>Sorbitol (D-Glucitol)</p>	$\xrightarrow{s(17), x(1,6)}$	$\begin{array}{ccccccc} & \text{OH} & \text{H} & \text{OH} & & & \\ &   &   &   & & & \\ \text{CH}_2\text{OH} & -\text{C} & -\text{C} & -\text{C} & -\text{CO} & -\text{CH}_2\text{OH} & \\ &   &   &   & & & \\ & \text{H} & \text{OH} & \text{H} & & & \end{array}$ <p>L-Sorbose</p>
$\begin{array}{ccccccc} & \text{OH} & \text{OH} & \text{OH} & \text{OH} & & \\ &   &   &   &   & & \\ \text{CH}_2\text{OH} & -\text{C} & -\text{C} & -\text{C} & -\text{C} & -\text{CH}_2\text{OH} & \\ &   &   &   &   & & \\ & \text{H} & \text{H} & \text{H} & \text{H} & & \end{array}$ <p>Allitol (<i>meso</i>)</p>	$\xrightarrow{x(34)}$	$\begin{array}{ccccccc} & \text{OH} & \text{OH} & \text{OH} & & & \\ &   &   &   & & & \\ \text{CH}_2\text{OH} & -\text{C} & -\text{C} & -\text{C} & -\text{CO} & -\text{CH}_2\text{OH} & \\ &   &   &   & & & \\ & \text{H} & \text{H} & \text{H} & & & \end{array}$ <p>L-Allulose (L-Psicose)</p>
$\begin{array}{ccccccc} & \text{H} & \text{OH} & \text{OH} & \text{H} & & \\ &   &   &   &   & & \\ \text{CH}_2\text{OH} & -\text{C} & -\text{C} & -\text{C} & -\text{C} & -\text{CH}_2\text{OH} & \\ &   &   &   &   & & \\ & \text{OH} & \text{H} & \text{H} & \text{OH} & & \end{array}$ <p>Dulcitol (<i>meso</i>)</p>	$\xrightarrow{s(14, 22), x(2)}$	No oxidation
$\begin{array}{ccccccc} & \text{H} & \text{OH} & \text{H} & \text{OH} & & \\ &   &   &   &   & & \\ \text{CH}_2\text{OH} & -\text{C} & -\text{C} & -\text{C} & -\text{C} & -\text{CH}_2\text{OH} & \\ &   &   &   &   & & \\ & \text{OH} & \text{H} & \text{OH} & \text{H} & & \end{array}$ <p>D-Iditol</p>	$\xrightarrow{x(6)}$	No oxidation
$\begin{array}{ccccccc} & \text{H} & \text{OH} & \text{OH} & \text{OH} & & \\ &   &   &   &   & & \\ \text{CH}_2\text{OH} & -\text{C} & -\text{C} & -\text{C} & -\text{C} & -\text{CH}_2\text{OH} & \\ &   &   &   &   & & \\ & \text{OH} & \text{H} & \text{H} & \text{H} & & \end{array}$ <p>D-Talitol</p>	$\xrightarrow{?}$	$\begin{array}{ccccccc} & \text{H} & \text{OH} & \text{OH} & & & \\ &   &   &   & & & \\ \text{CH}_2\text{OH} & -\text{C} & -\text{C} & -\text{C} & -\text{CO} & -\text{CH}_2\text{OH} & \\ &   &   &   & & & \\ & \text{OH} & \text{H} & \text{H} & & & \end{array}$ <p>D-Tagatose (predicted)</p>

Information on the oxidation of five heptitols and two octitols which have been tested is given in Table 7. The first four compounds in the table have the *cis* configuration and are oxidized, while the fifth does not possess the favorable configuration and is not oxidized, again confirming Bertrand's rule. The first two heptitols listed have been tested with both *A. xylinum* and *A. suboxydans*. Bertrand (7,8) isolated the ketoses formed by *A. xylinum*. Hann, Tilden, and Hudson (22) were the first to report the oxidation of these compounds by *A. suboxydans*. The L-perseulose was isolated by Tilden (35) and the structure and configuration proven

TABLE 7  
OXIDATION OF HEPTITOLS AND OCTITOLS

$\begin{array}{ccccccc} & \text{OH} & \text{H} & \text{H} & \text{OH} & \text{OH} & \\ \text{CH}_2\text{OH} & -\text{C}- & \text{C}- & \text{C}- & \text{C}- & \text{C}- & \text{CH}_2\text{OH} \\ & \text{H} & \text{OH} & \text{OH} & \text{H} & \text{H} & \\ & \text{D-Manno-D-gala-heptitol} & & & & & \\ & \text{(D-Perseitol)} & & & & & \end{array}$	$\xrightarrow{s(22,35), x(7)}$	$\begin{array}{ccccccc} & \text{OH} & \text{H} & \text{H} & \text{OH} & & \\ \text{CH}_2\text{OH} & -\text{C}- & \text{C}- & \text{C}- & \text{C}- & \text{CO}- & \text{CH}_2\text{OH} \\ & \text{H} & \text{OH} & \text{OH} & \text{H} & & \\ & \text{L-Gala-D-fructo-heptose} & & & & & \\ & \text{(L-Perseulose, L-Galaheptulose)} & & & & & \end{array}$
$\begin{array}{ccccccc} & \text{OH} & \text{OH} & \text{H} & \text{OH} & \text{OH} & \\ \text{CH}_2\text{OH} & -\text{C}- & \text{C}- & \text{C}- & \text{C}- & \text{C}- & \text{CH}_2\text{OH} \\ & \text{H} & \text{H} & \text{OH} & \text{H} & \text{H} & \\ & \text{Gluco-gulo-heptitol (meso)} & & & & & \\ & \text{(\alpha-Glucoheptitol)} & & & & & \end{array}$	$\xrightarrow{s(22), x(8)}$	$\begin{array}{ccccccc} & \text{OH} & \text{OH} & \text{H} & \text{OH} & & \\ \text{CH}_2\text{OH} & -\text{C}- & \text{C}- & \text{C}- & \text{C}- & \text{CO}- & \text{CH}_2\text{OH} \\ & \text{H} & \text{H} & \text{OH} & \text{H} & & \\ & \text{L-Gluco-L-sorbo-heptose} & & & & & \\ & \text{(L-Glucoheptulose)} & & & & & \end{array}$
$\begin{array}{ccccccc} & \text{H} & \text{H} & \text{OH} & \text{OH} & \text{OH} & \\ \text{CH}_2\text{OH} & -\text{C}- & \text{C}- & \text{C}- & \text{C}- & \text{C}- & \text{CH}_2\text{OH} \\ & \text{OH} & \text{OH} & \text{H} & \text{H} & \text{H} & \\ & \text{D-Manno-D-talo-heptitol} & & & & & \\ & \text{(D-Volemitol)} & & & & & \end{array}$	$\xrightarrow{x(2)}$	<p>"Volemulose"</p> <p>D-Manno-D-tagato-heptose and/or D-Altro-D-fructo-heptose</p>
$\begin{array}{ccccccc} & \text{H} & \text{OH} & \text{H} & \text{OH} & \text{OH} & \\ \text{CH}_2\text{OH} & -\text{C}- & \text{C}- & \text{C}- & \text{C}- & \text{C}- & \text{CH}_2\text{OH} \\ & \text{OH} & \text{H} & \text{OH} & \text{H} & \text{H} & \\ & \text{D-Gluco-D-ido-heptitol} & & & & & \\ & \text{(D-β-Glucoheptitol)} & & & & & \end{array}$	$\xrightarrow{x(12)}$	$\begin{array}{ccccccc} & \text{H} & \text{OH} & \text{H} & \text{OH} & & \\ \text{CH}_2\text{OH} & -\text{C}- & \text{C}- & \text{C}- & \text{C}- & \text{CO}- & \text{CH}_2\text{OH} \\ & \text{OH} & \text{H} & \text{OH} & \text{H} & & \\ & \text{D-Ido-L-sorbo-heptose} & & & & & \\ & \text{(D-Idoheptulose) (predicted)} & & & & & \end{array}$
$\begin{array}{ccccccc} & \text{H} & \text{OH} & \text{OH} & \text{H} & \text{OH} & \\ \text{CH}_2\text{OH} & -\text{C}- & \text{C}- & \text{C}- & \text{C}- & \text{C}- & \text{CH}_2\text{OH} \\ & \text{OH} & \text{H} & \text{H} & \text{OH} & \text{H} & \\ & \text{D-Gala-1-gluco-heptitol} & & & & & \\ & \text{(D-β-Galaheptitol)} & & & & & \end{array}$	$\xrightarrow{s(22)}$	No oxidation
$\begin{array}{ccccccc} & \text{OH} & \text{OH} & \text{OH} & \text{H} & \text{OH} & \text{OH} \\ \text{CH}_2\text{OH} & -\text{C}- & \text{C}- & \text{C}- & \text{C}- & \text{C}- & \text{C}- & \text{CH}_2\text{OH} \\ & \text{H} & \text{H} & \text{H} & \text{OH} & \text{H} & \text{H} & \\ & \text{D-Gluco-L-talo-octitol} & & & & & & \\ & \text{(D-α, β-Glucooctitol)} & & & & & & \end{array}$	$\xrightarrow{s(22)}$	$\begin{array}{ccccccc} & \text{OH} & \text{OH} & \text{OH} & \text{H} & \text{OH} & \\ \text{CH}_2\text{OH} & -\text{C}- & \text{C}- & \text{C}- & \text{C}- & \text{C}- & \text{CO}- & \text{CH}_2\text{OH} \\ & \text{H} & \text{H} & \text{H} & \text{OH} & \text{H} & & \\ & \text{L-Altro-L-sorbo-octose} & & & & & & \\ & \text{(predicted)} & & & & & & \end{array}$
$\begin{array}{ccccccc} & \text{H} & \text{OH} & \text{OH} & \text{H} & \text{H} & \text{OH} \\ \text{CH}_2\text{OH} & -\text{C}- & \text{C}- & \text{C}- & \text{C}- & \text{C}- & \text{CH}_2\text{OH} \\ & \text{OH} & \text{H} & \text{H} & \text{OH} & \text{OH} & \text{H} & \\ & \text{D-Gala-1-gala-octitol} & & & & & & \\ & \text{(D-α, α-galaooctitol)} & & & & & & \end{array}$	$\xrightarrow{s(22)}$	No oxidation

by Hann and Hudson (21). The oxidation product of gluco-gulo-heptitol was not isolated by Hann, Tilden, and Hudson (22), but it can be assumed that the ketose is identical with that isolated and identified by Bertrand as produced by the action of *A. xylinum*. This conclusion is justified by the fact that in all previous cases in which the oxidation products have been identified, the same ketoses have been formed by both organisms. This assumption will be taken for granted in subsequent discussion.

The third and fourth heptitols shown in Table 7 were tested only with *A. xylinum* and the oxidation products were not identified. Since

D-manno-D-talo-heptitol has the favorable *cis* arrangement on both ends of the molecule, it can be predicted that the reaction would give D-manno-D-tagato-heptose or D-altro-D-fructo-heptose or a mixture of the two. Bertrand designated the products simply as "volemulose." It can also be predicted that the oxidation of D-glucio-D-ido-heptitol would give D-ido-L-sorbo-heptose.

The last three polyhydric alcohols of Table 7 were tested by Hann, Tilden, and Hudson (22). The reducing sugar formed from the D-glucio-L-talo-octitol was not isolated or identified but the formation of L-altro-L-sorbo-octose can be predicted.

*Configuration Required for the Oxidation of the Sugar Alcohols Having More than Four Carbon Atoms.* Previous discussion has demonstrated conformity to Bertrand's rule that a *cis* arrangement of the two secondary alcohol groups next to the primary alcohol group is required

TABLE 8  
D-CONFIGURATION NECESSARY FOR OXIDATION OF SUGAR ALCOHOLS

CH <sub>2</sub> OH— $\begin{array}{c} \text{H} \quad \text{OH} \quad \text{OH} \\   \quad   \quad   \\ \text{C} - \text{C} - \text{C} \\   \quad   \quad   \\ \text{OH} \quad \text{H} \quad \text{H} \end{array}$ —CH <sub>2</sub> OH	$\xrightarrow{s(22)}$	CH <sub>2</sub> OH— $\begin{array}{c} \text{H} \quad \text{OH} \\   \quad   \\ \text{C} - \text{C} \\   \quad   \\ \text{OH} \quad \text{H} \end{array}$ —CO—CH <sub>2</sub> OH
D-Arabitol		D-Xylulose
CH <sub>2</sub> OH— $\begin{array}{c} \text{OH} \quad \text{H} \quad \text{H} \\   \quad   \quad   \\ \text{C} - \text{C} - \text{C} \\   \quad   \quad   \\ \text{H} \quad \text{OH} \quad \text{OH} \end{array}$ —CH <sub>2</sub> OH	$\xrightarrow{s(22)}$	No oxidation
L-Arabitol		
CH <sub>2</sub> OH— $\begin{array}{c} \text{OH} \quad \text{H} \quad \text{H} \quad \text{OH} \quad \text{OH} \\   \quad   \quad   \quad   \quad   \\ \text{C} - \text{C} - \text{C} - \text{C} - \text{C} \\   \quad   \quad   \quad   \quad   \\ \text{H} \quad \text{OH} \quad \text{OH} \quad \text{H} \quad \text{H} \end{array}$ —CH <sub>2</sub> OH	$\xrightarrow{s(22), x(7)}$	CH <sub>2</sub> OH— $\begin{array}{c} \text{OH} \quad \text{H} \quad \text{H} \quad \text{OH} \\   \quad   \quad   \quad   \\ \text{C} - \text{C} - \text{C} - \text{C} \\   \quad   \quad   \quad   \\ \text{H} \quad \text{OH} \quad \text{OH} \quad \text{H} \end{array}$ —CO—CH <sub>2</sub> OH
D-Perseitol		L-Perseulose
CH <sub>2</sub> OH— $\begin{array}{c} \text{H} \quad \text{OH} \quad \text{OH} \quad \text{H} \quad \text{H} \\   \quad   \quad   \quad   \quad   \\ \text{C} - \text{C} - \text{C} - \text{C} - \text{C} \\   \quad   \quad   \quad   \quad   \\ \text{OH} \quad \text{H} \quad \text{H} \quad \text{OH} \quad \text{OH} \end{array}$ —CH <sub>2</sub> OH	$\xrightarrow{s(22)}$	No oxidation
L-Perseitol		
CH <sub>2</sub> OH— $\begin{array}{c} \text{OH} \quad \text{OH} \\   \quad   \\ \text{C} - \text{C} \\   \quad   \\ \text{H} \quad \text{H} \end{array}$ —CH <sub>2</sub> OH	$\xrightarrow{s(22, 42), x(5)}$	CH <sub>2</sub> OH— $\begin{array}{c} \text{OH} \\   \\ \text{C} \\   \\ \text{H} \end{array}$ —CO—CH <sub>2</sub> OH
meso-Erythritol		L-Erythrulose
CH <sub>2</sub> OH— $\begin{array}{c} \text{OH} \quad \text{OH} \quad \text{OH} \\   \quad   \quad   \\ \text{C} - \text{C} - \text{C} \\   \quad   \quad   \\ \text{H} \quad \text{H} \quad \text{H} \end{array}$ —CH <sub>2</sub> OH	$\xrightarrow{s(32)}$	CH <sub>2</sub> OH— $\begin{array}{c} \text{OH} \quad \text{OH} \\   \quad   \\ \text{C} - \text{C} \\   \quad   \\ \text{H} \quad \text{H} \end{array}$ —CO—CH <sub>2</sub> OH
Adonitol (meso)		L-Adonulose
CH <sub>2</sub> OH— $\begin{array}{c} \text{OH} \quad \text{OH} \quad \text{OH} \quad \text{OH} \\   \quad   \quad   \quad   \\ \text{C} - \text{C} - \text{C} - \text{C} \\   \quad   \quad   \quad   \\ \text{H} \quad \text{H} \quad \text{H} \quad \text{H} \end{array}$ —CH <sub>2</sub> OH	$\xrightarrow{s, x(34)}$	CH <sub>2</sub> OH— $\begin{array}{c} \text{OH} \quad \text{OH} \quad \text{OH} \\   \quad   \quad   \\ \text{C} - \text{C} - \text{C} \\   \quad   \quad   \\ \text{H} \quad \text{H} \quad \text{H} \end{array}$ —CO—CH <sub>2</sub> OH
Allitol (meso)		L-Allulose

for the oxidation of the sugar alcohols by members of the genus *Acetobacter*. Hann, Tilden, and Hudson (22) investigated the action of *A. suboxydans* upon a number of sugar alcohols, including two pairs of enantiomorphs; their results with the latter are included in Table 8. It is evident that while D-arabitol and D-perseitol are readily oxidized their enantiomorphs are not attacked. Hann, Tilden and Hudson properly concluded that the organism is so specific that the *cis* pair of secondary alcohol groups must have the D-configuration. The fact that only ketoses of L-configuration are obtained in high yields from the *meso*-sugar alcohols shown in Table 8 confirms this generalization. That *A. suboxydans* is more specific than is *A. xylinum* is further evidenced by the report of Bertrand (5) that L-arabitol is oxidized by the latter organism. Further evidence on the greater specificity of *A. suboxydans* will be presented below. However, no exception has been reported, in which oxidation products have been conclusively identified, to this extraordinarily high specificity of *A. suboxydans*.

#### OXIDATION OF ERYTHRITOL AND THE GLYCOLS

Data are presented in Table 9 on the oxidation of several polyhydric alcohols having only two secondary alcohol groups. Both *A. xylinum* and *A. suboxydans* produce L-erythrulose from *meso*-erythritol. This compound has the favorable *cis* arrangement of the secondary alcohol groups. *Meso*-2,3-butanediol, which is similar to *meso*-erythritol except that it has two terminal methyl groups is oxidized to L-(+)-acetylmethylcarbinol by *A. suboxydans*. It has been found in our laboratories, however, that the organism also oxidizes the D-(−)-2,3-butanediol but does not attack the L-(+)-2,3-butanediol. That is, with glycols containing the terminal methyl groups, the *cis* arrangement of the secondary alcohol groups is not required. It would be of great interest to test the action of the organism on D- and L-erythritol to determine whether similar results would be obtained with terminal primary alcohol groups.

Grivsky (20) confirmed our findings with the 2,3-butanediols using *A. xylinum* and *A. aceti*. However, on prolonged incubation, after complete conversion of the D-2,3-butanediol from a racemic mixture had occurred, the L-diol was slowly oxidized. This was not the case with *A. suboxydans*. This again illustrates the high specificity of the latter organism.

The results shown in Table 9 for the 3,4-hexanediols were obtained by Van Risseghem (38) employing *A. xylinum* and *A. aceti*. With the hexanediols, as with the butanediols, the secondary alcohol group having the D-configuration is preferentially attacked. On the basis of their results, Van Risseghem (38) and Grivsky (20) were able to assign the proper configurations to the 3,4-hexanediols and the 2,3-butanediols. Grivsky's configurations for the butanediols, deduced from microbiological procedure, agree with the configurations assigned to them, on the basis of chemical methods, by Morell and Auernheimer (26).

On the basis of the glycols studied, it may be concluded that secondary

TABLE 9  
 OXIDATION OF ERYTHRITOL AND GLYCOLS

$\begin{array}{c} \text{CH}_2\text{OH}-\text{C}-\text{C}-\text{CH}_2\text{OH} \\   \quad   \\ \text{H} \quad \text{H} \end{array}$ <i>meso</i> -Erythritol	$\xrightarrow{s(42), x(5), a(23)}$	$\begin{array}{c} \text{CH}_2\text{OH}-\text{C}-\text{CO}-\text{CH}_2\text{OH} \\   \\ \text{H} \end{array}$ <i>L</i> -Erythrulose
$\begin{array}{c} \text{CH}_3-\text{C}-\text{C}-\text{CH}_3 \\   \quad   \\ \text{H} \quad \text{H} \end{array}$ <i>meso</i> -2,3-Butanediol	$\xrightarrow{s(19, 37), x(20), a(20)}$	$\begin{array}{c} \text{CH}_3-\text{C}-\text{CO}-\text{CH}_3 \\   \\ \text{H} \end{array}$ <i>L</i> -(+)-Acetylmethylcarbinol
$\begin{array}{c} \text{CH}_3-\text{C}-\text{C}-\text{CH}_3 \\   \quad   \\ \text{OH} \quad \text{H} \end{array}$ <i>D</i> -(-)-2,3-Butanediol	$\xrightarrow{s(37), x(20), a(20)}$	$\begin{array}{c} \text{CH}_3-\text{C}-\text{CO}-\text{CH}_3 \\   \\ \text{OH} \end{array}$ <i>D</i> -(-)-Acetylmethylcarbinol
$\begin{array}{c} \text{CH}_3-\text{C}-\text{C}-\text{CH}_3 \\   \quad   \\ \text{H} \quad \text{OH} \end{array}$ <i>L</i> -(+)-2,3-Butanediol	$\xrightarrow{s(37), x(20), a(20)}$	No oxidation
$\begin{array}{c} \text{CH}_3\text{CH}_2-\text{C}-\text{C}-\text{CH}_2\text{CH}_3 \\   \quad   \\ \text{H} \quad \text{H} \end{array}$ <i>meso</i> -3,4-Hexanediol	$\xrightarrow{x(38), a(38)}$	$\begin{array}{c} \text{CH}_3\text{CH}_2-\text{C}-\text{CO}-\text{CH}_2\text{CH}_3 \\   \\ \text{H} \end{array}$ <i>L</i> -(+)-Ethylpropionylcarbinol
$\begin{array}{c} \text{CH}_3\text{CH}_2-\text{C}-\text{C}-\text{CH}_2\text{CH}_3 \\   \quad   \\ \text{OH} \quad \text{H} \end{array}$ <i>D</i> -(+)-3,4-Hexanediol	$\xrightarrow{x(38), a(38)}$	$\begin{array}{c} \text{CH}_3\text{CH}_2-\text{C}-\text{CO}-\text{CH}_2\text{CH}_3 \\   \\ \text{OH} \end{array}$ <i>D</i> -(-)-Ethylpropionylcarbinol
$\begin{array}{c} \text{CH}_3\text{CH}_2-\text{C}-\text{C}-\text{CH}_2\text{CH}_3 \\   \quad   \\ \text{H} \quad \text{OH} \end{array}$ <i>L</i> -(-)-3,4-Hexanediol	$\xrightarrow{x(38), a(38)}$	No oxidation

alcohol groups of *D*-configuration are required for oxidation with *Acetobacter suboxydans* and that those of *L*-configuration are not attacked. It was previously noted that Hann, Tilden, and Hudson (22) came to the conclusion that Bertrand's rule is followed only in case the *cis* secondary alcohol groups have the *D*-configuration. With the glycols, only the secondary alcohol group with *D*-configuration is oxidized but the *cis* arrangement is not required.

#### OXIDATION OF THE DESOXY SUGAR ALCOHOLS

In Table 10 are shown results of tests reported on the oxidation of several desoxy sugar alcohols by members of the genus *Acetobacter*. All are desoxy in the terminal position except 2-desoxy-*D*-sorbitol. In an oral paper presented at the 110th Meeting of the American Chemical Society,

TABLE 10  
OXIDATION OF DESOXY SUGAR ALCOHOLS

$\begin{array}{ccccccc} & \text{OH} & \text{OH} & \text{H} & \text{H} & & \\ \text{CH}_3 & -\text{C}- & \text{C}- & \text{C}- & \text{C}- & \text{CH}_2\text{OH} & \\ & \text{H} & \text{H} & \text{OH} & \text{OH} & & \end{array}$ <p>L-Rhamnitol</p>	$\xrightarrow{s(14, 22), x(39)}$	No oxidation
$\begin{array}{ccccccc} & \text{H} & \text{OH} & \text{OH} & \text{H} & & \\ \text{CH}_3 & -\text{C}- & \text{C}- & \text{C}- & \text{C}- & \text{CH}_2\text{OH} & \\ & \text{OH} & \text{H} & \text{H} & \text{OH} & & \end{array}$ <p>D-Fucitol (Rhodeitol)</p>	$\xrightarrow{x(39)}$	No oxidation
$\begin{array}{ccccccc} & \text{OH} & \text{H} & \text{H} & \text{OH} & & \\ \text{CH}_3 & -\text{C}- & \text{C}- & \text{C}- & \text{C}- & \text{CH}_2\text{OH} & \\ & \text{H} & \text{OH} & \text{OH} & \text{H} & & \end{array}$ <p>L-Fucitol</p>	$\xrightarrow{s(22)}$	Reducing compound (unidentified)
$\begin{array}{ccccccc} & \text{OH} & \text{OH} & \text{H} & \text{H} & \text{OH} & \\ \text{CH}_3 & -\text{C}- & \text{C}- & \text{C}- & \text{C}- & \text{C}- & \text{CH}_2\text{OH} \\ & \text{H} & \text{H} & \text{OH} & \text{OH} & \text{H} & \end{array}$ <p>1-Manno-L-gala-7-desoxy-heptitol (α-Rhamnohexitol)</p>	$\xrightarrow{x(39)}$	No oxidation
$\begin{array}{ccccccc} & \text{OH} & \text{OH} & \text{H} & \text{H} & \text{H} & \\ \text{CH}_3 & -\text{C}- & \text{C}- & \text{C}- & \text{C}- & \text{C}- & \text{CH}_2\text{OH} \\ & \text{H} & \text{H} & \text{OH} & \text{OH} & \text{OH} & \end{array}$ <p>L-Manno-1-talo-7-desoxy-heptitol (β-Rhamnohexitol)</p>	$\xrightarrow{x(39)}$	No oxidation
$\begin{array}{ccccccc} & \text{H} & \text{OH} & \text{OH} & & & \\ \text{CH}_2\text{OH} & -\text{CH}_2- & \text{C}- & \text{C}- & \text{C}- & \text{CH}_2\text{OH} & \\ & & \text{OH} & \text{H} & \text{H} & & \end{array}$ <p>2-Desoxy-D-sorbitol</p>	$\xrightarrow{s}$	$\begin{array}{ccccccc} & \text{H} & \text{OH} & & & & \\ \text{CH}_2\text{OH} & -\text{CH}_2- & \text{C}- & \text{C}- & \text{CO}- & \text{CH}_2\text{OH} & \\ & & \text{OH} & \text{H} & & & \end{array}$ <p>5-Desoxy-L-sorbose</p>

Regna reported that this compound was oxidized by *A. suboxydans* to 5-desoxy-L-sorbose which was isolated and characterized. The oxidative reactions with the desoxy sugar alcohols follow Bertrand's rule of necessary *cis* secondary alcohol groups as well as the required *D*-configuration except in the case of L-fucitol which apparently breaks both rules. Hann, Tilden, and Hudson (22) found that *A. suboxydans* converted L-fucitol to a reducing substance which was not isolated or identified. We thoroughly agree with these authors in their statement that: "The behavior of L-fucitol must be studied further before deciding what generalization may apply to the alcohols derived from the methylose sugars."

#### SUMMARY OF STEREOCHEMICAL RELATIONSHIP

Stereochemical relations have been presented for the oxidation of 31 polyhydric alcohols by members of the genus *Acetobacter*. With possible exception of L-fucitol, the following generalizations can be made with



reference to the specificity of *A. suboxydans* in the oxidation of straight chain sugar alcohols:

1. Only the 2-keto compounds are formed from sugar alcohols having terminal primary alcohol groups.

2. The secondary alcohol group oxidized must possess the D-configuration.

3. The polyhydric alcohols having more than two secondary alcohol groups must have *cis* arrangement as well as D-configuration.

It is evident that *A. suboxydans* furnishes a remarkably specific catalytic tool for the production of keto compounds from the polyhydric alcohols and in the elucidation of the stereochemistry and configuration of these compounds. Only a small part of this field has been adequately explored.

#### SUMMARY

Species of the genus *Acetobacter*, especially *Acetobacter suboxydans*, oxidize certain polyhydric alcohols to produce ketose sugars. This method enables the production on laboratory or commercial scale of ketoses otherwise obtainable only with great difficulty. Production of sorbose, levulose, dihydroxyacetone, erythrulose, a keto-inositol and acetylmethylcarbinol are reviewed, detailed procedures being given for the laboratory preparation of dihydroxyacetone and the keto-inositol. Stereochemical considerations are presented which lead to the conclusion that *A. suboxydans* furnishes a remarkably specific catalytic tool for production of keto compounds from the polyhydric alcohols and in the elucidation of the configurations of these compounds.

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# STUDIES ON OXYGEN-CARRYING COBALT COMPOUNDS

## I. OXYGEN-CARRYING METALLO-ORGANIC COMPOUNDS. THEIR USE IN THE MANUFACTURE OF OXYGEN

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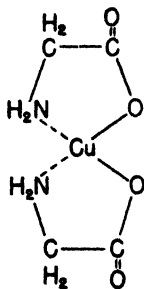
One of the most striking aspects of biochemistry is the use nature has made of small quantities of metals in promoting and controlling the various complicated chemical processes of life. Hemoglobin, utilizing iron in the transport of oxygen by the blood of the mammals, and chlorophyll, utilizing magnesium in the reduction of carbon dioxide by the plants, are the more widely appreciated of these natural, metal-organic materials, but equally intriguing are the copper, manganese, and vanadium oxygen-carriers in the blood of the lower animals and the metal-containing enzymes governing certain biological, oxidation-reduction processes. The well-being of many plants and animals is dependent on the proper functioning of these metallo-organic compounds and on the availability of minute amounts of other metals, particularly zinc and cobalt, whose action is exerted through coordination compounds with protein. An abundant literature already exists describing the effects of deficiencies of these metals, but only a start has been made in elucidating the chemistry of the metallo-organic compounds involved.

The linkage of the metal to the organic molecule in these compounds is through the functional groups of the organic molecules, no case of a direct linkage of carbon to metal being known. The functional group may be an acidic group, in which case the metal replaces a hydrogen atom, or it may be a basic group, to which the metal is attached by a so-called secondary or coordinate valence. When the functional group involved is an acidic group the bond may be ionic or non-ionic in character, that is, the metal may be split off as an ion or held closely to the organic group in a non-ionic or covalent form. The attachment to a basic group is non-ionic in character. In the usual case more than one functional group is present in the organic molecule and a combination of both types of valences is involved. Frequently there is also involved the formation of rings or cages in which the metal is implicated in the ring or cage structure. Such ring structures often possess extraordinary thermal stability; the compounds usually have colors departing widely from the customary colors of the metal salts, and they frequently are soluble in non-polar solvents and insoluble in water.

The composition and structure of the numerous compounds of ammonia and the metals, particularly of cobalt and platinum, puzzled chemists for a century. It was the elucidation of the nature of these

compounds which has supplied the tools for attacking the problems of the naturally occurring metallo-organic compounds. In 1893 the Swiss chemist Alfred Werner devised the coordination theory which explained the composition of the metal ammoniates on the basis of secondary valence, a new type of valence bond by which apparently saturated compounds were able to unite to form new compounds. Coupling this new concept of valence with a stereochemical explanation of the structure of the compounds, Werner was able to organize the ammoniates into a single system and to correlate an enormous mass of chemical information. Werner's studies culminated in 1912 in the optical resolution of a purely inorganic compound which unequivocally established the coordination theory and secured for its author the Nobel Prize. During the subsequent two decades the coordination theory was expanded and applied to a variety of chemical problems, notably by Paul Pfeiffer in Germany and by G. T. Morgan in England. The coordination theory touched many fields of chemistry but it was particularly fruitful in its application to the dyeing of textiles with metallic mordants, to the discovery of new organic analytical reagents, to leather tanning, and to the study of the stereochemical nature of the metal ions.

That ring formation was involved in the union of metals with functional organic compounds was recognized simultaneously by Bruni and Fornara (1) and by Ley (2) in 1904 while studying the bright blue copper compound of glycine, to which they ascribed the structure

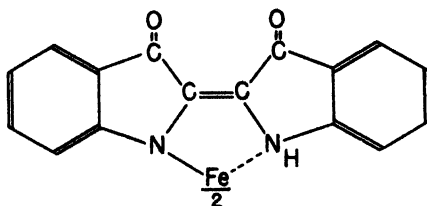


In the following years the conditions necessary for ring formation of this type were pointed out and subsequently numerous compounds, new and old, were shown to possess such a cyclic structure. Drew and Morgan (3) coined the term *chelate ring*, derived from the claw of the lobster and crustaceans, for these cyclic metal-containing compounds. Later Morgan devised the terms *bidentate* and *quadridentate* to distinguish those compounds in which the metal was attached to the organic molecule through two functional organic groups from those in which the attachment was through four groups. The field of the chelate rings has been carefully reviewed in recent years and the reader's attention is directed to a more detailed treatment of this field than is possible here (4).

Although the physiological behavior and the chemical constitution of hemoglobin have been extremely well worked out as a result of the

classical researches of Barcroft (5), Fischer (6), and many others, the nature of the mechanism by which the oxygen becomes attached to the hemoglobin molecule is still uncertain. Indeed, even the nature of the linkage by which heme, the porphyrine part of the molecule, is attached to the protein, or globin, part of the molecule is not known. Fischer, who is largely responsible for the knowledge of the chemical constitution of hemoglobin, has practically nothing to say regarding the attachment of the heme to the globin or the nature of the union of hemoglobin with oxygen. Of the great deal of speculation found in the literature as to the nature of the hemoglobin-oxygen linkage, probably the most reasonable is that of Wahl who has attempted to apply the Werner coordination theory to the problem (7). Reasoning by analogy with certain cobalt-ammonia compounds which are known to absorb oxygen in such a manner as to release it on acidification, Wahl proposed a similar arrangement for hemoglobin. The oxygen enters these cobalt compounds as a bivalent, acidic peroxo group,  $-O-O-$ . Similar, simple, peroxo compounds of iron are not known, however. On theoretical grounds Wahl was able to build up a hypothesis to account for the change in acidity of hemoglobin on absorbing oxygen which is related to the equally important function of transporting carbon dioxide in the reverse direction. From a stereochemical standpoint the Wahl hypothesis is weak.

The closest approach to hemoglobin of a synthetic compound capable of carrying oxygen reversibly is the iron-indigo compound of Kunz and Kress (8), synthesized by the reaction of iron carbonyl with indigo in a pyridine solution. Carbon monoxide was evolved and a yellowish-red compound was obtained containing one atom of iron per molecule of indigo.



A pyridine solution of this compound absorbed oxygen, the color shifting at the same time from red to green. One molecule of oxygen was absorbed for each atom of iron, and on the application of a vacuum the oxygen was evolved and the color reverted to the original red. Carbon monoxide destroyed the ability of the solution to absorb oxygen. After several cycles this ability to absorb and release oxygen was lost owing to a gradual, irreversible oxidation of the organic material.

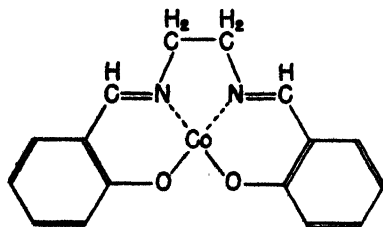
Hemoglobin has never been used for the manufacture of oxygen, although the patent of Sinding-Larsen (9) indicates that attempts have been made to do so. The body, of course, has mechanisms whereby the hemoglobin is constantly regenerated and it would appear that the rapid deterioration of the hemoglobin would make any *in vitro* process impracticable. The Kunz and Kress compound also cannot be used in this

manner owing to the rapid, irreversible oxidation of the material. Other inorganic compounds have been used, of course, for the recovery of oxygen from the atmosphere, barium oxide being the most noteworthy. The barium oxide process was investigated in some detail toward the close of the last century by the Brin brothers and others. It received a great deal of attention in the years around the turn of the century but was completely abandoned by 1920 in favor of the liquid air process. Considering the vastly improved engineering materials now available and the information regarding the conditioning of air now at hand, there is reason to believe that the Brin process might well operate economically in competition with the liquid air process. The principal disadvantage of the Brin process is the necessity of maintaining a considerably elevated temperature, of the order of  $600^{\circ}$ . The advantages of a chemical which would reversibly absorb and release oxygen at room temperature are obvious.

Bivalent cobalt salts in the presence of ammonia have the property of absorbing large quantities of oxygen. There is formed during this absorption a polynuclear coordination compound having a peroxo linkage between two cobalt atoms,  $\text{Co-O-O-Co}$ . On acidification the oxygen is expelled from this material and it is possible to use it for the recovery of oxygen from the atmosphere. A critical study of the process was made by Gluud, Keller, and Nordt (10) who concluded that such a process could never succeed commercially—principally because of irreversible oxidation of some cobalt to the trivalent state during each cycle. Warne and Woolcock (11) have applied the process to the manufacture of oxygen, using hexamminocobaltous perchlorate and a somewhat different technique; the oxygen was first caused to unite with the complex cobalt compound with the simultaneous expulsion of ammonia and then expelled from the peroxo cobalt compound so formed with the simultaneous reunion with ammonia. The equilibrium was shifted to and fro by controlling the concentration of ammonia, a mechanism for doing this being described. This process is indeed ingenious but there is no record of its actual utilization commercially.

Numerous binuclear cobalt ammoniates have been described by Werner and others (12), but a detailed examination covering many of these compounds has failed to reveal any in which the compounds are reported to reversibly absorb and release oxygen by a change in temperature or pressure.

The organic cobalt compound disalicylalethylenediimine cobalt first



mentioned by Pfeiffer, Breith, Lübke, and Tsumaki (13) and later studied in more detail by Tsumaki (14), is quite unique in this respect. Tsumaki found that it absorbed about 3.5 per cent in weight of oxygen, corresponding more or less to a ratio of cobalt to an oxygen molecule of three to one. He found that the oxygen was expelled from the compound by heating it to 100° in a stream of carbon dioxide. Although it would appear from the work of Tsumaki that the compound once it had absorbed and given off its oxygen might be used again in the same manner, a careful reading of the Tsumaki papers discloses that he apparently did not appreciate the significance of such a cyclic behavior or prove that it was possible. It is strange that Tsumaki failed to grasp the significance of the material as he failed to mention either the possibility of using it for the recovery of oxygen from the atmosphere or of discovering means whereby the oxygen could be expelled and collected without the use of carbon dioxide.

The work reported in this series of papers is devoted almost entirely to this particular compound and to materials derived from it in various ways. The work was directed specifically to the utilization of the material for the commercial production of oxygen and many of the aspects of the problem of academic importance were deferred for future investigation. Broadly, the phases of the problem which were investigated and are being reported are: (1) the satisfactory manufacture of the material on a large scale, (2) the exact chemical constitution and the structure of the material, (3) the physical properties with particular reference to the ability to absorb and release oxygen, (4) the modification of the physical properties of the compound by the introduction of substituents, (5) the methods whereby it can be used for the generation of oxygen, and (6) the effective life and the economics of its utilization in the manufacture of oxygen.

Extensive studies were made of the methods of preparing the oxygen-carrying compound (designated Co-Ox) especially for its large-scale manufacture. The proper conditions were found for eliminating the side reactions, of securing consistently a material having the maximum oxygen-carrying capacity, of increasing the yield, and of decreasing the cost. There are several ways in which this cobalt compound can be made and the number of factors affecting the quality of the product is surprisingly large. These are dealt with in detail in Paper II.

Paper III deals with the chemical and physical properties of disalicylalethylenediimine cobalt. The exact composition of the material was determined as far as the difficulty of purifying the material permitted. It was discovered that water is present in the molecule in the ratio of one molecule of water to two cobalt atoms. Various proofs of the presence of this water are presented, including its direct determination by means of the Karl Fischer reagent and the synthesis under anhydrous conditions (Paper IV) of an inactive, orange material which becomes the oxygen carrier when treated with water. Apparently the water molecule acts as a bridging group to hold the two cobalt atoms, each surrounded by the quadridentate, chelating molecule of disalicylalethylenediimine, together



in a binuclear compound. The compound is thus actually bi-(disalicylalethylenediimine)- $\mu$ -aquo-dicobalt. There are two vacant positions in this compound, properly oriented to each other to permit the absorption of oxygen to form a peroxo group. The mechanism and stereochemistry of this are discussed in Paper III.

A red, inactive isomer of bi-(disalicylalethylenediimine)- $\mu$ -aquo-dicobalt was also found and the conditions for its formation and conversion to the active isomer are reported. Bi-(disalicylalethylenediimine)- $\mu$ -aquo-dicobalt was found to absorb nitric oxide and nitrogen dioxide also but not carbon monoxide and other gases. The compound is paramagnetic and becomes diamagnetic on absorbing oxygen. The rate of oxygenation of bi-(disalicylalethylenediimine)- $\mu$ -aquo-dicobalt at various temperatures and oxygen pressures was determined. The apparatus used for such measurements on this and the other compounds studied is described in Paper XIII and the data on the parent compound is presented in Paper III.

Numerous related compounds were prepared from substituted salicylaldehydes. Of the few which possessed the property of reversibly absorbing and releasing oxygen, those from 3-nitrosalicylaldehyde, 3-methoxysalicylaldehyde, 3-ethoxysalicylaldehyde and 3-*n*-butoxysalicylaldehyde absorbed oxygen more rapidly than the parent compound and were made the subject of more detailed studies, Papers V, VI, and VII. A few other compounds were prepared which were active toward oxygen, notably those derived from the various methylsalicylaldehydes, 3-chlorosalicylaldehyde, and *o*-hydroxyacetophenone, but the rate at which they absorbed oxygen was low. These compounds and the compounds which were found to be inactive are described in Paper VIII.

Substitution in or for the ethylenediamine portion of the molecule of disalicylalethylenediimine invariably produced a cobalt compound inactive toward oxygen. A variety of other diamines (Paper IX) and monamines (Paper X) were tried, all without success.

An interesting group of oxygen-carrying materials was obtained by starting with mixtures of aldehydes, for example salicylaldehyde and 3-methoxysalicylaldehyde. The physical properties of the cobalt derivatives depart considerably from those of the materials from the pure aldehydes owing to mixed crystal formation. This work is described in Paper XI. In Paper XII it is shown that an unsymmetrical Schiff's base of ethylenediamine cannot be made.

The concluding paper of the series deals with the engineering aspects of the manufacture of oxygen using these compounds. The compound may be used in a stationary bed or it may be used continuously by circulating the solid oxygen carrier from an oxygenation chamber to a deoxygenation chamber and back. Experience with each type of operation is reported in Paper XIV.

Several difficulties arose in the utilization of the material in the manufacture of oxygen from air. The material underwent a deep-seated, irreversible oxidation, deteriorating to about 50 per cent of its original, oxygen-carrying capacity in five thousand cycles. This is far too rapid

to be economic. The material is a light, fluffy powder which is an excellent heat insulator; the oxygenation-deoxygenation reaction is accompanied by a large heat of reaction, of the order of 20,000 calories per mole of oxygen. Consequently heat transfer during the cyclic operation becomes a major factor in the design of equipment. The material undergoes a change in density on oxygenation and the continued working of the crystal in cyclic operation, combined with its somewhat greasy, graphitic surface character, cause a stationary bed of the material to set up to a very hard solid which makes its replacement in the reaction vessel difficult. The light, fluffy powder is exceptionally fine, is difficult to filter and quite toxic. The opinion at present is that the process cannot be a serious competitor of the liquid air process. Nor do any of the substituted materials give promise of longer life or better thermal conductivity.

Academically the problem is of great interest. The mechanism of the oxygen absorption must be different from that of hemoglobin and oxygen, since in the latter one molecule of oxygen is absorbed per atom of iron while in the cobalt compound the ratio is one molecule of oxygen per two atoms of cobalt. This is, however, sufficient resemblance of the material to biologically important metal-containing materials to make its further study of value.

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# STUDIES ON OXYGEN-CARRYING COBALT COMPOUNDS

## II. THE METHODS OF PREPARING

### BI-(DISALICYLALETHYLENEDIIMINE)- $\mu$ -AQUO-DICOBALT, CO-OX

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The compound, bi-(disalicylaethylenediimine)- $\mu$ -aquo-dicobalt, was first prepared by bringing together simultaneously in an aqueous-alcohol reaction medium, cobalt acetate, ethylenediamine and salicylaldehyde (1). Somewhat later the same procedure was used and the product recrystallized from benzene and chloroform (2).

Our preliminary studies indicated that if care was used in the preparation, the oxygen-carrying capacity of the product could be greatly raised from the value of 3.5 per cent first reported. The formation of the cinnamon-colored, oxygen-carrying compound was complicated by the formation under certain conditions of three by-products, olive green, black, and red in color, all of which were found to be inactive toward oxygen.

The olive by-product was formed on prolonged contact of the cinnamon-colored, oxygen-carrying material with hot water, either during the formation of the material or during its drying. When heated at 200° in a vacuum it was partially reconverted to the active compound. It was also formed when the filtrate from a preparation of the cinnamon-colored, active compound was evaporated. The presence of any free acid, especially when hot, promoted the formation of the olive by-product.

The black by-product was formed from the cinnamon-colored, oxygen-carrying compound by contact with oxygen in the presence of alcohol. The black compound could not be deoxygenated. By washing the oxygen-carrying compound with dilute alcohol, or better with water, the formation of the black by-product was minimized.

The red by-product resulted from the action of alkali on the cinnamon-colored, oxygen-carrying compound. It was found to be isomeric with the oxygen-carrying compound but inactive toward oxygen. This compound is discussed in more detail in Paper III.

The theoretical value for the oxygen-carrying capacity of Co-Ox is 4.79 per cent, that is  $\frac{32}{2(325) + 18}$ , 325 being the molecular weight of the cobalt-organic portion of the material and 18 being the molecular weight of the molecule of water associated with each two cobalt atoms (see Paper III for a discussion of this). Material of capacity greater than 4.7 per cent is therefore quite satisfactory. Somewhat higher capacities were found in certain instances; this was probably due to the adsorption

of oxygen on the finely powdered, oxygen-carrying material since the excess seldom exceeded 0.2 per cent and was rapidly lost at atmospheric pressure.

The earlier method of preparing this material was that of reacting simultaneously, solutions of cobalt acetate, ethylenediamine, and salicylaldehyde, in the molecular ratio of 1, 1, and 2, respectively, this reaction being most easily effected in water and alcohol so that the final solution contained about 60 per cent alcohol. It is possible in this procedure to form, in addition to Co-Ox, other compounds, principally cobalt salicylaldehyde, cobalt ethylenediamine salts, and the Schiff's base, disalicylalethylenediimine. Unless care was exercised the product was relatively impure. It was possible, however, by controlling a number of factors properly, to prepare a material having a high oxygen-carrying capacity by this method and the method was satisfactory for the preparation of the material in large quantities. Purer material was obtained by first preparing the Schiff's base, disalicylalethylenediimine, a beautifully crystalline, bright yellow solid. This Schiff's base was dissolved either in alkali or in alcohol and treated with an aqueous solution of a cobalt salt, yielding the same product as before but in a purer state.

Besides securing the stoichiometric ratio of the reactants, it was necessary in order to obtain a good product of high oxygen-carrying capacity to secure optimum conditions with respect to certain other factors, principally the rate at which the reactants were mixed, the volume of solvent used, the nature of the solvent, the temperature, the time of standing, the exposure to air while standing, the effectiveness of the washing after filtration, the speed at which the material was dried, and the temperature of drying. All of these factors were investigated and are discussed below.

For convenience the two methods just discussed are designated *Method A* (direct mixing of all three reactants) and *Method B* (preliminary preparation of Schiff's base).

Obviously it was desirable to eliminate the use of alcohol in the preparation. Numerous experiments with *Method A*, the direct mixing method, showed that when carried out without alcohol, the product was always of low oxygen-carrying capacity. *Method B* could, however, be carried out without alcohol and a satisfactory product obtained. The Schiff's base, disalicylalethylenediimine, was made by the addition of salicylaldehyde to a diluted, aqueous solution of ethylenediamine. To a solution of this yellow Schiff's base in a sodium hydroxide solution was then added a solution of a cobalt salt. It was found essential that the solution contain somewhat less than the two molecules of sodium hydroxide theoretically required since the alkali produced by hydrolysis of the salt of the Schiff's base or any excess sodium hydroxide added caused the formation of the red, inactive form of disalicylalethylenediimine cobalt.

It was desirable to use a cheaper salt of cobalt than the acetate, for example, the chloride, nitrate, or sulfate. When using a cobalt salt of a strong acid it was necessary to neutralize the strong acid liberated,

otherwise the cobalt compound was formed only to the extent of 20 to 30 per cent. The neutralization was made with sodium acetate or with sodium hydroxide or carbonate; with the latter two it was essential that a deficiency be used to prevent the formation of the inactive, red isomer.

In the interests of conserving space the numerous experiments which led to the conclusions stated above will not be described, but each of the various factors involved in the preparation are discussed in more detail and the working directions given for the best procedures developed.

A third method, designated *Method C*, was also of some interest. In it the pyridine addition compound, a red, crystalline solid, was first prepared and then rendered active toward oxygen by expelling the pyridine by heating the material in a vacuum. This procedure did not yield a product of high oxygen-carrying capacity and the method is impracticable for the preparation of large amounts of material.

It was observed that on the addition of a solution of a cobalt salt to a solution of the Schiff's base, disalicylaethylenediimine, there was first formed an orange precipitate which quickly changed over to the cinnamon-colored oxygen-carrier. This orange compound was isolated in pure form by carrying out the reaction under anhydrous conditions; it is discussed in detail in Paper IV.

#### FACTORS INVOLVED IN THE PREPARATION OF Co-Ox IN PURE FORM

Using Method A or Method B satisfactory material can only be prepared if certain factors are closely controlled.

##### FACTOR 1.

In some of our earlier work the Co-Ox produced was badly contaminated by the olive-colored by-product, even when the salicylaldehyde was distilled prior to use. It was found that the commercial salicylaldehyde contained some hydrochloric acid which distilled with the aldehyde. This acid was easily eliminated by treating the aldehyde with solid sodium bicarbonate or calcium carbonate prior to distillation. When distilled salicylaldehyde free from hydrochloric acid was employed, no trouble from this source was encountered.

The olive-colored material is produced by the prolonged action of water on Co-Ox particularly in the presence of free acid.

##### FACTOR 2.

In Method A it was possible to introduce three contaminating products by departing from strictly equivalent amounts of the reactants or by working in such a manner that the three reactants did not come together at the same time. Thus cobalt and salicylaldehyde formed a yellow, insoluble compound under about the same conditions in which Method A was carried out. Again, ethylenediamine and cobalt salts reacted to give compounds which contaminated the preparation. On the other hand, ethylenediamine and salicylaldehyde gave the yellow Schiff's

base which was only slightly soluble (about 4 per cent) in cold alcohol and less soluble in aqueous-alcohol mixtures. It was evident, therefore, that if the proper amounts of the reactants were not used, contamination would arise from three sources. A little consideration of this immediately showed that it was particularly important that the ethylenediamine and salicylaldehyde be added in exactly the ratio of one to two and that at least enough cobalt be present to react with the amine and aldehyde. A slight excess of cobalt acetate did no harm and was even desirable since the excess of cobalt acetate remained in solution and was subsequently washed out.

When the reaction was carried out observing these considerations, no further trouble from this source was encountered.

The neatest way found to insure the correct ratio of ethylenediamine to salicylaldehyde was to first prepare the yellow condensation product of the two, as is done in Method B. This, however, introduced another step.

Closely connected with the excess of the reagents is the matter of the order in which the reagents are added. Obviously, in Method A, since any two of the reagents may react to cause a contamination, it is necessary to get all of the reagents mixed as rapidly as possible. When following Method A it was found best to add the ethylenediamine and the salicylaldehyde, both previously diluted with about their own volume of 60–70 per cent alcohol, simultaneously to the cobalt acetate solution, stirring with all the vigor and effectiveness possible, and to maintain the stirring until the mass had set up to a thick paste, a matter of about 45 seconds.

In Method B it was most convenient to add the hot, aqueous solution of cobalt acetate to a hot, alcohol solution of the condensation product. The alcohol solution was cooled slightly as otherwise violent boiling occurred when the aqueous solution was added. The addition was carried out as rapidly as possible with effective stirring or shaking.

### FACTOR 3.

The temperature of the solutions at the time of mixing in Method A had apparently little effect as preparations made with hot solutions were no better in yield or oxygen-carrying capacity than those made in the cold. In Method B the reaction was practically limited to hot solutions because of the relative insolubility of disalicylaethylenediimine in cold alcohol.

### FACTOR 4.

When using water-alcohol mixtures as the reaction medium the oxygen-carrying capacity dropped when the alcohol concentration was made above 60 per cent. Under strictly anhydrous conditions other compounds are formed which yield Co-Ox on treatment with water (Paper IV). In addition, the formation of the black by-product on contact with air became more serious at the higher alcohol concentrations.

## FACTOR 5.

If the volume of the aqueous-alcohol mixture used as solvent was too large the compound failed to form completely and the yield fell. In order to minimize the loss of product because of this, the volume of the solvent mixture was decreased as much as feasible. On the other hand, decreasing the volume too far caused the formation of contaminating products which decreased the oxygen-carrying capacity. In Method A the best condition found was that volume of 60 per cent alcohol which would just hold the required cobalt acetate in solution at room temperature. This required about three liters of the mixture per gram mole of cobalt acetate. The volume of alcohol used to dissolve the other two reagents then was small in comparison to the volume used to dissolve the cobalt acetate and did not sufficiently change the composition of the solvent to be of consequence.

In Method B it was found most convenient to dissolve the condensation product in 95 per cent alcohol. Attempts were made to dilute this solution of the condensation product with water to give a solvent mixture containing 60–70 per cent alcohol. This was done very carefully in order to prevent the precipitation of the yellow condensation product. By dissolving the cobalt acetate in that volume of water, which on addition to the solution of the condensation product in 95 per cent alcohol gave a mixture containing about 60–70 per cent alcohol, excellent results were obtained. On adding the aqueous solution of cobalt acetate (heated to a temperature of about 95°) to the alcohol solution of the condensation product cooled slightly below its boiling point, some cinnamon-colored material was formed immediately and the remainder precipitated within a minute or two. Since the product was obtained in high yield and of high capacity, the local concentration of the alcohol at the point of precipitation must have been about that necessary to give the cinnamon-colored material without any of the red by-product; apparently also the hot water raised the temperature of the solvent so that no yellow condensation product was thrown out to contaminate the cinnamon-colored material.

Attempts to isolate more of the oxygen-carrying compound from the filtrate after centrifuging off the cinnamon-colored material failed, owing to the formation of the olive-colored compound during the evaporation.

## FACTOR 6.

Contact with air at the time of the reaction in methods A or B or during the period of standing before centrifuging lead to the formation of the black by-product, probably a trivalent cobalt compound. This material was soluble in water and alcohol and passed into the filtrate on filtering and washing. It was formed only when the material was still wet with the aqueous-alcohol mixture. The formation of this black material did not affect the yield appreciably, but its formation was minimized by sweeping the oxygen out of the reactants before mixing and by

maintaining an atmosphere of an inert gas over the reaction mixture during the period of standing before centrifuging. It was found equally satisfactory to cover the reaction mixture with a layer of aqueous-alcohol during the period of standing.

FACTOR 7.

The period of standing after the reaction and before centrifuging had little effect on the yield or oxygen-carrying capacity of the product. About fifteen minutes were required for the completion of the reaction; further standing did no harm. Thus, preparations filtered after fifteen minutes were identical with preparations which stood one hour, five hours, overnight, or one week.

FACTOR 8.

In Method A the reaction was usually carried out at room temperature and no advantage accrued from running the reaction at higher temperatures. The temperature rose about 15° during the reaction. The temperature fell during the period of standing so that cooling was not necessary. Since in Method B the reaction was carried out hot, the mixture was cooled before centrifuging. It was not necessary to cool this reaction before filtering; in fact, filtering hot was faster and aided in drying.

FACTOR 9.

As mentioned under Factor 6, a black by-product was formed when the cinnamon-colored material came in contact with air while still wet with alcohol. Washing with aqueous-alcohol mixtures further promoted the conversion of the material to this black by-product. It was found much better to wash immediately with water rather than with aqueous-alcohol mixtures. Water washed out the black material and left a uniform cinnamon-colored material, which was stable toward air.

FACTOR 10.

It was found that the washing of the precipitate could not be effectively carried out on the centrifuge. The cake was therefore removed from the centrifuge basket, broken up, stirred with wash water until a uniform slurry was produced, and the mixture again centrifuged. It was found best to repeat this a second time.

Continued washing with water did not finally give a clear filtrate. A reaction between the water and compound took place slowly giving the olive by-product which passed into the filtrate.

FACTOR 11.

One of the most important factors in the preparation of the oxygen-carrying, cinnamon-colored material is that of drying. Small preparations made in early, small scale, laboratory studies were dried in a steam heated, Fischer type, vacuum drying pistol. Larger amounts of material



were dried in a vacuum drying oven consisting of a horizontal brass tube, 12 inches in diameter and 20 inches long, enclosed in an electrically heated oven. When material was placed in this oven more than a half inch deep, the drying was very slow and the preparations were generally of low oxygen-carrying capacity. As mentioned under Factor 10 a reaction between water and the oxygen-carrying compound occurred. This reaction was slow at room temperature but was much more rapid when the wet compound was hot. During the slow drying process a considerable portion of the cinnamon-colored material was converted to the olive material and the oxygen-carrying capacity greatly decreased.

In another attempt to dry the centrifuged and washed compound, a stream of warm, dried carbon dioxide was passed over the compound. Although this worked fairly well it was still too slow to be satisfactory.

For handling one pound batches of compound, four glass tubes 1.25 inches in diameter and 36 inches long were finally used. These were set up vertically, surrounded by heating jackets and evacuated with an oil pump, properly protected with condenser, trap, and drying train. The material was broken up and placed in the tubes in small pieces. A total of one pound of material could be dried in this manner in three to four hours. The water was pulled off quite rapidly and the compound underwent very slight alteration during this time.

The drying process was materially assisted by pressing the water from the freshly filtered product. Using a hydraulic press and a pressure of about one ton per square inch, some 70 per cent of the moisture in the cake was expelled. Only a short time was necessary then for the vacuum drying and the oxygen-carrying capacity of the product was very high.

It was found possible to dry the cake at atmospheric pressure by exposing thin layers to 250 watt, reflector type drying bulbs at a distance of about six inches. This procedure worked best when most of the water was removed from the cake by pressure. The oxygen-carrying capacity never exceeded 4.5 per cent when the material was dried in this manner.

#### PREFERRED METHODS FOR THE PREPARATION OF Co-Ox

##### METHOD A. DIRECT MIXING OF REACTANTS

In a 1.5 gallon crock place 250 g. (1 mole plus 0.9 g. excess) of cobalt acetate,  $\text{Co}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 4\text{H}_2\text{O}$  and 3 l. of 60 per cent ethyl alcohol, sp. gr. 0.91. Agitate until all of the salt has dissolved. If a clear solution does not result it should be filtered. Prepare a mixture of 100 ml. of ethyl alcohol and 60.1 g. (1 mole) of ethylenediamine by weighing out that amount of aqueous ethylenediamine solution needed (ethylenediamine is marketed as an aqueous solution containing about 70 per cent ethylenediamine). Prepare a mixture of 244.2 g. of salicylaldehyde and approximately 250 ml. of alcohol (if any doubt exists about the purity of the salicylaldehyde it should first be treated with solid calcium carbonate, filtered, and then distilled). With a broad paddle agitate the cobalt acetate solution as vigorously as possible, and add the ethylenediamine solution. Add immediately and as rapidly as possible the salicylaldehyde

solution, maintaining the stirring at a vigorous rate. Continue to stir vigorously until the mixture sets up to a reddish-brown paste. Cover the mass with a half inch layer of water and allow to stand at least fifteen minutes.

Centrifuge off the solid and continue centrifuging until the mother liquor is completely removed. Add three portions of about 250 ml. of water, allowing the liquid to be centrifuged off each time before making the next addition. Remove the cake from the centrifuge basket and mix it up thoroughly with 1.5 l. of water so that no large particles remain and a uniform slurry is obtained. Filter again by centrifuging. Remove the cake and wash again in the same manner. Finally centrifuge as dry as possible. The filtrate will never run through clear but will be light brown in color. Break up the cake into small pieces, arrange in thin layers and dry at  $100^{\circ}$  in a good vacuum.

#### METHOD B. FROM THE SCHIFF'S BASE

*Preparation of Disalicylalethylenediimine.* Dissolve 244 g. (2 moles) of salicylaldehyde in 1 l. of boiling, 95 per cent ethyl alcohol. Stir and add 60.1 g. (1 mole) of ethylenediamine, measured by weighing out that amount of aqueous ethylenediamine solution needed. In 20–30 seconds the mass becomes solid with a bright yellow, crystalline material. Cool the reaction mixture, and filter on a Buchner funnel. The product may be spread out in thin layers on absorbent paper to dry. It may be recrystallized from 6 l. of hot 95 per cent alcohol or used without further purification. It will dissolve somewhat more rapidly in the next step if not allowed to dry out. Yield: about 255 g. or 95 per cent.

*Preparation of Co-Ox, Using Alcohol.* Dissolve 268 g. (1 mole) of disalicylalethylenediimine in 10 l. of boiling 95 per cent alcohol. In another vessel dissolve 250 g. (1 mole plus 0.9 g. excess) of cobalt acetate,  $\text{Co}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 4\text{H}_2\text{O}$ , in 1.5 l. of boiling water; filter if a clear solution does not result. Turn off all burners. Cool the alcohol solution slightly below its boiling point, and with vigorous stirring, pour the hot cobalt acetate solution into the alcohol solution as rapidly as permissible. Vigorous boiling occurs and a considerable volume of alcohol vapors are evolved. Some compound forms immediately, and the reaction mixture sets up to a red-brown, pasty solid after about ten minutes. Cool the mixture below  $30^{\circ}$ . Continue with the filtration and remaining operations as described in the second paragraph of the procedure of Method A. Yield: 270 g., 80 per cent; oxygen-carrying capacity 4.7 to 4.8 per cent.

*Preparation of Co-Ox, Without Using Alcohol.* Dissolve 268 g. (1.0 mole) of finely ground disalicylalethylenediimine, 79.5 g. (2.0 moles less 0.5 g.) of sodium hydroxide, and 5 g. of sodium acetate,  $\text{NaC}_2\text{H}_3\text{O}_2 \cdot 3\text{H}_2\text{O}$ , in 3 l. of boiling water. The solution of the disalicylalethylenediimine requires from ten to twenty minutes and depends on the state of subdivision of the material and the agitation given the mixture. When the solution is complete, except possibly for the presence of a small amount of yellow scum on the surface of the solution, add to the solution 238 g. (1.0 mole

of cobalt chloride  $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ ) dissolved in 500 ml. of hot water. Agitate the solution vigorously during the addition of the cobalt salt solution. Continue with the filtration and remaining operations as described in the second paragraph of the procedure of Method A. Yield: 300 g., 90 per cent; oxygen-carrying capacity: 4.7 to 4.8 per cent.

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2. Tsumaki, *Bull. Chem. Soc. Japan*, 13:252. 1938.

# STUDIES ON OXYGEN-CARRYING COBALT COMPOUNDS

## III. THE COMPOSITION AND CHEMICAL PROPERTIES OF

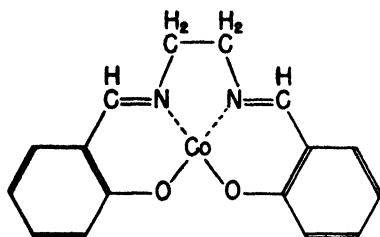
### BI-(DISALICYLALETHYLENEDIIMINE)- $\mu$ -AQUO-DICOBALT, CO-OX

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Obviously nothing in the simple composition of disalicylaethylenediimine cobalt predicates or explains the remarkable behavior of the



compound in reversibly absorbing and releasing oxygen. Since the oxygen and the cobalt combine in the ratio of one molecule of oxygen to two atoms of cobalt and because of the improbability of the oxygen molecule becoming dissociated in the process, it is likely that the attachment of the oxygen molecule to the cobalt is as a peroxo group

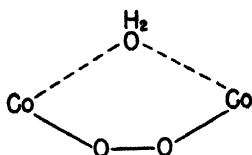


As indicated in Paper I of this series, compounds of this type are well known among the coordination compounds of cobalt, the peroxo group functioning as a bridging group to hold together the cobalt atoms of these so-called "polynuclear compounds." Usually in the polynuclear compounds containing peroxo bridging groups there is present a second or third bridging group, a hydroxyl, amino, or nitro group.

Disalicylaethylenediimine occupies four of the coordination positions of the cobalt atom. Cobalt, however, invariably has the coordination number six. It becomes pertinent, therefore, to inquire if all of the coordination positions are not filled in this compound, if perhaps there is not also present in the molecule a bridging group, water or possibly hy-

<sup>1</sup> The work by Diehl and Chao was done at Purdue University during the fall of 1938.

droxyl, tying the cobalt atoms together and arranging a convenient place for the oxygen molecule to enter to form a peroxo bridge:



That water is necessary for the formation of the oxygen-carrying compound is shown in Paper IV dealing with the reaction of disalicylaethylenediimine and cobalt chloride under anhydrous conditions. Under anhydrous conditions an orange material, inactive toward oxygen, is formed which yields the oxygen-carrying compound on treatment with water. The presence of the water in the compound could not be proved or disproved by chemical analysis owing to the lack of a satisfactory method of recrystallizing the compound for purification. Nor was a solvent found in which the molecular weight could be determined by the freezing or boiling point methods. Direct proof of the presence of the water was obtained, however, by heating the material in anhydrous pyridine, collecting the distillate and determining by means of the Karl Fischer reagent the water expelled from the compound and distilled over with the pyridine. Each of these aspects of the problem are discussed in some detail in the following sections.

#### ANALYSIS AND COMPOSITION

Unfortunately Co-Ox is insoluble in water and most organic solvents and cannot be recrystallized for purification. It is soluble in pyridine and chloroform but crystallizes from these solvents with solvent of crystallization in which form it does not absorb oxygen. On removal of the solvent of crystallization by heating the material in a vacuum only part of the activity toward oxygen is restored. Thus, the compound must be prepared sufficiently pure for analysis when first formed. Using the best methods of preparation found (Paper II of this series) and using nickel-free cobalt salts, several highly pure specimens of Co-Ox were made. The oxygen-carrying capacities of these materials ranged from 4.70 to 4.80 per cent, agreeing well with the theoretical value of 4.79 for a compound possessing a half molecule of water per cobalt atom.

These materials were analyzed for cobalt, nitrogen, carbon, and hydrogen as carefully as possible. The results on a single preparation were in excellent agreement, but minor variations were found from preparation to preparation indicating that an absolutely pure compound was not obtained. On one preparation the molecular weight as calculated from the analysis for cobalt was 326, from the analysis for nitrogen 335, carbon 332, and hydrogen (assuming 14 hydrogen atoms present)

328. On another sample the analysis for cobalt indicated 332 and for nitrogen 328. On a third the analysis for nitrogen gave 332. The theoretical molecular weight of the anhydrous molecule,  $C_{16}H_{14}O_2N_2Co$ , is 325, that of a hemihydrate 334, and that of molecule containing a hydroxyl group as a bridging group ( $\mu$ -hydroxyl) 333.5. The results indicate that some other material is present but the uncertainty in the analytical results from preparation to preparation is just sufficient to prohibit drawing a categorical conclusion. The analysis of one sample for hydrogen gave: 4.32, 4.27, 4.33, 4.36, 4.28, aver.: 4.30. This indicated that no hydrogen was present beyond that required by the simple, anhydrous formulation, 4.31 per cent, significantly below a  $\mu$ -hydroxyl material which would contain 4.35 per cent hydrogen or a hemihydrate 4.49 per cent hydrogen.

#### MOLECULAR WEIGHT

It is obvious that the question of the binuclear composition of the compound could be settled positively if the molecular weight of the material could be determined. Disalicylalethylenediimine cobalt is somewhat soluble in chloroform and in pyridine. It crystallizes from these solvents with chloroform or pyridine of crystallization so that in solution the material undoubtedly ties up some solvent. In a cryoscopic determination of molecular weight in these solvents this would be negligible with respect to the total volume of solvent or could be corrected for on the basis of one molecule of solvent for each cobalt. However, in both chloroform and in pyridine by the boiling point method, a depression of the boiling point was obtained instead of the expected elevation. This can only be explained on the basis of water being expelled from the compound and increasing the vapor pressure.

An apparatus was constructed for measuring the lowering of the freezing point of pyridine. It consisted of two cells, one for pure pyridine and the second for the pyridine solution, a ten junction thermopile, mechanical stirrers in each cell and in the surrounding toluene bath which was contained in a large Dewar flask. The bath was cooled by dry ice. Suitable radiation shields were inserted. A White potentiometer was used to measure the potential of the thermopile. This apparatus worked very well with naphthalene and the Schiff's base, disalicylalethylenediimine. When the cobalt compound was dissolved in the pyridine, however, reproducible and steady values could not be obtained for the freezing point. It appeared again that water was being drawn from the compound even in the cold, assisted undoubtedly by the preferential coordination of pyridine to the cobalt forming a compound containing two molecules of pyridine per cobalt atom.

The solubility of Co-Ox in a variety of solvents was determined roughly. The solubility in the three most promising solvents, benzene, ethylenedibromide, and bromoform, was too low to give a significant change in the freezing point or the boiling point.

## DIRECT DETERMINATION OF WATER PRESENT IN Co-Ox

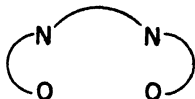
The most direct attack on the question of the presence of water in the oxygen-carrying compound appeared to be the expulsion of the water at a relatively high temperature and its gravimetric determination in a suitable desiccant. Experiments along this line indicated that such water if present was not expelled at temperatures up to  $190^{\circ}$ . Slow decomposition occurred above  $170^{\circ}$  with the liberation of salicylaldehyde. The formation of salicylaldehyde from the Schiff's base requires water but this can be considered only qualitative evidence.

The failure of the efforts to determine the molecular weight of the material cryoscopically provided the key to the solution of the problem. Since pyridine apparently expels the water from the compound it was only necessary to heat the material with pyridine and determine the water distilled with the pyridine. The water was determined by the Karl Fischer reagent. The results on one sample were 2.21 and 2.26 per cent water, on another sample 2.47 per cent; the calculated amount of water for a half molecule of water per cobalt atom is 2.69. The details of this work are described in the experimental section.

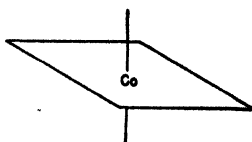
Polynuclear cobalt compounds of the type proposed here are well known (7) but the bridging groups previously reported have been the hydroxyl (-OH), the amino (-NH<sub>2</sub>), the nitro (-NO<sub>2</sub>), the peroxy (-O-O-), and the oxo (-O-). This appears to be the first case of a water molecule acting as the bridging group.

## STEREOCHEMISTRY

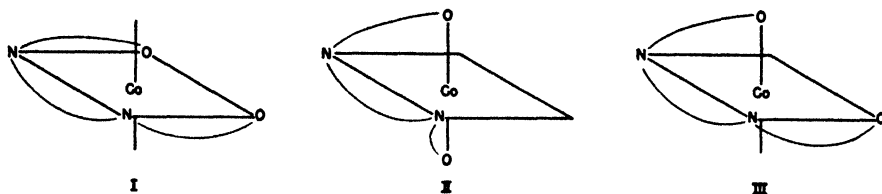
Having proved the presence of a half molecule of water per cobalt atom, the binuclear character of the compound is inescapable and it becomes of interest to inquire into the stereochemical arrangement of the various molecules about the cobalt atoms. There exist three ways in which the organic molecule, disalicylalethylenediimine, can be arranged about the cobalt atom. Adopting for the disalicylalethylenediimine molecule the shorter symbol



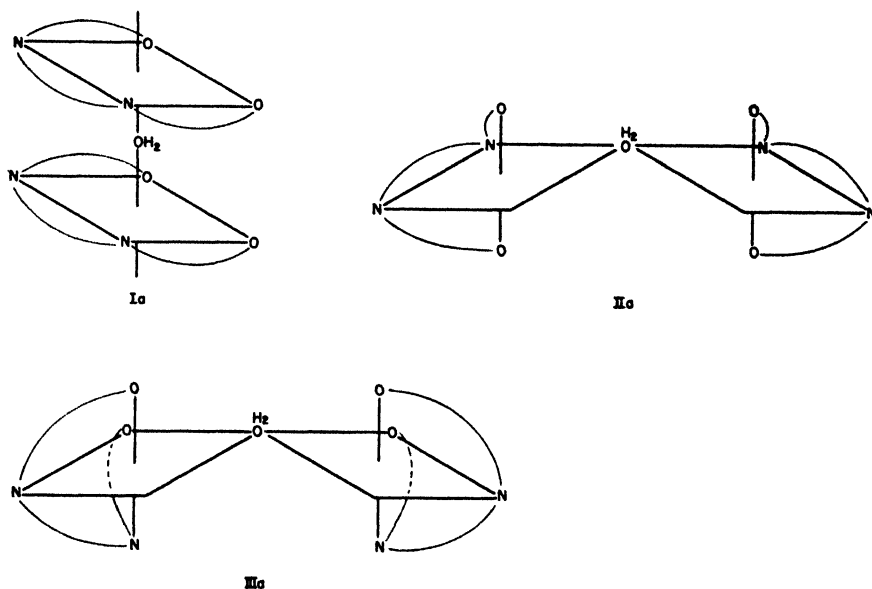
and utilizing the usual abbreviated octahedron for designating the six coordination positions about the cobalt atom



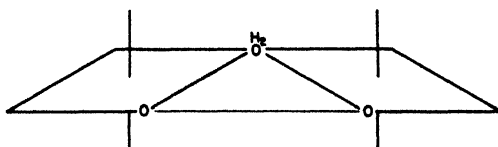
these arrangements are



All of these forms are more or less strain free as shown by models, but form I is perhaps the most stable. Considering the half molecule of water present per cobalt atom to function as a bridging group between two cobalt atoms, the structure of the various forms then becomes



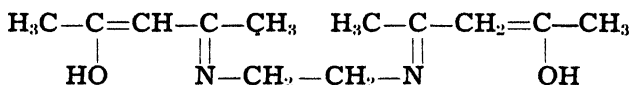
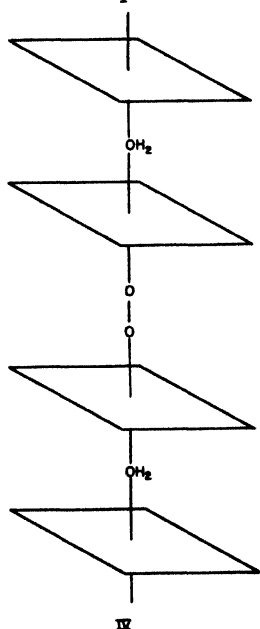
In each of the three structures, Ia, IIa, and IIIa, the sixth coordination position of each of the cobalt atoms is left vacant. In structures IIa and IIIa these positions are adjoining and there is just sufficient room for an oxygen molecule to slip into the empty space to form a peroxo bridge.



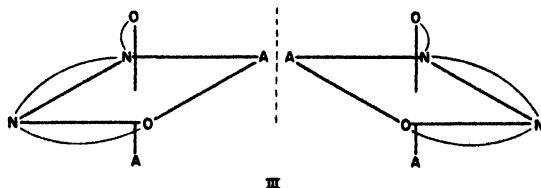
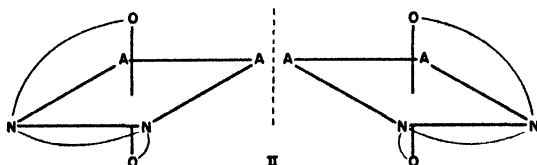
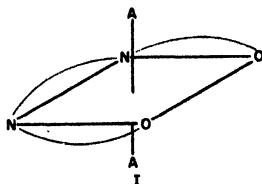


In structure Ia there is no such convenient space provided for the oxygen molecule and oxygen absorption could only then occur intermolecularly which requires that the crystal structure be so arranged that this may occur as in IV. This structure is much less probable for the oxygen-carrying compound than structures IIa and IIIa, in which the favorable orientations are forced by the *cis*-arrangement of the bridging water molecule and the vacant coordination positions made accessible to the oxygen molecule.

Similar isomeric compounds were prepared by Morgan and Smith (4) who prepared three isomeric forms of diacetylacetoethylenediimine cobalt, two of which they were able to resolve, owing to their unsymmetric nature. Representing the chelating compound



appropriately, these isomers corresponded to



A = NH<sub>3</sub>, Cl<sup>-</sup>, etc.

identical with the possible forms suggested for Co-Ox with the difference that in Co-Ox the aromatic rings lend less flexibility to the molecule.

On this basis there should exist three isomeric forms of bi- (disalicylal-ethylenediimine)- $\mu$ -aquo-dicobalt, two of which should carry oxygen and one of which should be inactive. As a result of X-ray diffraction and absorption spectra studies, it was shown that the material prepared in an aqueous medium from the Schiff's base is identical with the material prepared in an aqueous-alcohol medium by direct mixing of the reactants. The bright red, inactive compound discussed below is unquestionably one of the isomeric forms. A third form has not yet been found.

#### THE BRIGHT RED, INACTIVE ISOMER

As mentioned in Paper II of this series, a bright red, crystalline compound may be obtained in place of the cinnamon-colored, oxygen-carrying compound when an excess of alkali is present during the synthesis. It appeared desirable to obtain a pure preparation of this bright red material and to ascertain its character. A satisfactory procedure was devised for converting Co-Ox into the inactive isomer by heating it with alcoholic potassium hydroxide. Analyses indicated that the material was isomeric with Co-Ox and probably also contained a half molecule of water.

It was observed that the red compound was converted to the active form by grinding it with mineral oil and a crystalline material such as lucite or potassium chloride; if the mineral oil was washed out with benzene the material reverted to the inactive form.

X-ray diffraction (powder) patterns showed that Co-Ox and its red, inactive isomer are distinctly different compounds. No detailed crystallographic study of the material was attempted.

#### THE EFFECT OF GASES OTHER THAN OXYGEN ON Co-Ox

Co-Ox did not change in color or gain in weight when placed in carbon monoxide at atmospheric pressure. This is rather surprising in view of the extreme avidity of hemoglobin for carbon monoxide and of the Kunz and Kress iron-indigo compound for carbon monoxide.

Co-Ox absorbed nitric oxide rapidly with the evolution of heat. The compound turned dark blue in color and experienced a gain in weight corresponding to one molecule of nitric oxide per cobalt atom. The nitric oxide addition product was very stable; only about one-third of the nitric oxide was expelled at 170° in a vacuum. The bright red, inactive isomer of Co-Ox also absorbed nitric oxide and although the rate of absorption was much smaller, the total gain corresponded to about 1.35 molecules of nitric oxide per cobalt atom. The absorption of nitric oxide in this case was almost completely reversible.

In the case of nitrogen dioxide, the Schiff's base as well as Co-Ox and the inactive, red isomer absorbed nitrogen dioxide. All three materials absorbed approximately four molecules of gas per molecule of compound. Apparently the reaction in this case is different from the absorp-

tion of oxygen and nitric oxide, the nitrogen dioxide attacking the organic material, possibly at the double bond.

Nitrous oxide was not absorbed by Co-Ox or by its red isomer.

Deoxygenated Co-Ox is paramagnetic. It is quite possible therefore that the first step in the oxygenation of Co-Ox is a magnetic coupling of the cobalt compound with the oxygen molecule. It would be expected then that other paramagnetic gases should be absorbed by disalicylal-ethylenediimine cobalt but that diamagnetic gases should not be absorbed. The failure of the diamagnetic gases, carbon monoxide, carbon dioxide, nitrous oxide, nitrogen, and argon to be absorbed is in agreement with this. On the other hand, nitric oxide and nitrogen dioxide, which are paramagnetic, are absorbed, although the evidence for the latter is confused by the action of the nitrogen dioxide on the organic portion of the molecule.

Carbon monoxide can be rendered paramagnetic by radiation with ultra-violet light of very short wave length and chlorine can also be rendered paramagnetic by exposure to certain radiation; it would be of interest at some time in the future to study the absorption of these gases under such conditions.

Assuming that the process by which the oxygen is absorbed by bi-(disalicylal-ethylenediimine)- $\mu$ -aquo-dicobalt is the two step process just outlined, it would appear that the second step has a large temperature coefficient since the rate at which oxygen is absorbed decreases as the temperature decreases. The magnetic interaction, however, should become stronger at lower temperatures and it is predicted that oxygen will be absorbed by the compound at very low temperatures, the magnetic interaction of the oxygen and the cobalt compound being sufficient to hold the oxygen and cobalt compound together without the further step involving the formation of a peroxo bridge.

#### MAGNETIC SUSCEPTIBILITY AND ELECTRONIC STRUCTURE

The magnetic susceptibility of oxygenated and deoxygenated Co-Ox was measured. The deoxygenated form was found to be paramagnetic and the oxygenated form diamagnetic. The molar susceptibility using the molecular weight of 334 which is that of one cobalt atom surrounded by one molecule of disalicylal-ethylenediimine of the deoxygenated form was  $316 \times 10^{-5}$  corresponding to an effective moment 2.73 Bohr magnetons. This indicates one free electron per cobalt atom.

One unpaired electron per cobalt atom is what would be expected in this material. The atomic number of cobalt is 27, the cobalt is bivalent, and to the 25 electrons around the cobalt atom are added, eight electrons from the four covalent linkages with the organic material and two from the oxygen of the bridging water molecule, making a total of 35, the odd 4p electron remaining unpaired. The oxygen molecule is paramagnetic, having two unpaired electrons. The coupling, then, of the cobalt compound with the oxygen molecule is probably by the pairing of the un-

coupled electrons, two cobalt atoms being required to pair off the two unpaired electrons of the oxygen molecule.

#### RATE OF OXYGENATION

The rate of oxygenation of bi-(disalicylalethylenediimine)- $\mu$ -aquo-dicobalt was determined using the gas volumetric method described in Paper XIV, *Method D*. The material was carried on circular fins attached to a vertical metal tube through which cooling water passed; the heat transfer was therefore excellent. The purity of the oxygen used was determined by the usual gas analysis method using the alkaline hydro-sulfite reagent. Both cylinder oxygen and oxygen generated from potassium chlorate and manganese dioxide in a carefully evacuated generator were used. The retaining liquids in the apparatus were freed of air by boiling or by sweeping with oxygen and then protected from the air.

Using a constant temperature of 25–27°, the rate of oxygenation was determined at various pressures from 200 to 870 mm. of mercury. As will be seen from Figure 1, the rate of oxygenation decreased rapidly with pressure, and the per cent of oxygen taken up at infinite time, taken as four hours, also decreased. The point of inflection of the curves about corresponded to half saturation, and the time of half saturation values were therefore taken as a typical point for the comparison of the rates at different pressures. A plot of these times against pressure is given in Figure 2.

The rate of oxygenation was measured at various temperatures from  $-7^{\circ}$  to  $+41^{\circ}$ , holding the pressure constant at 510 mm. of mercury. As will be seen from the results which are presented graphically

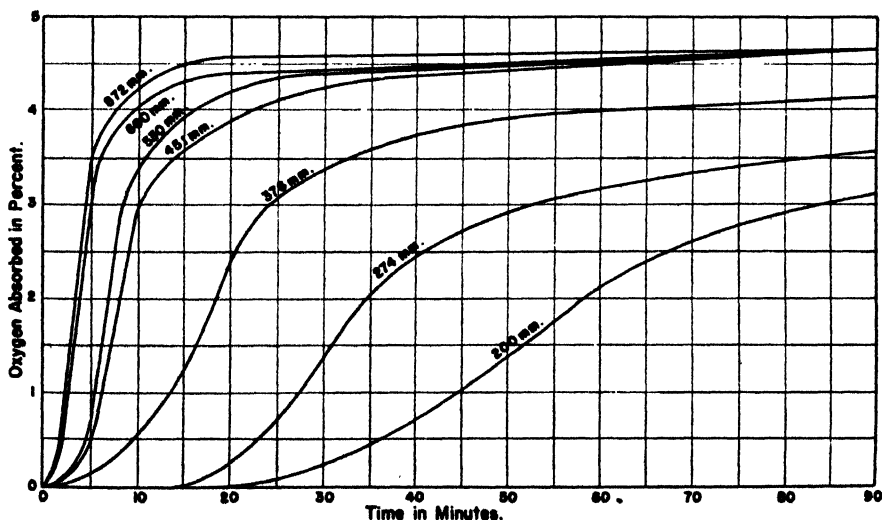


FIG. 1. Rate of oxygenation of bi-(disalicylalethylenediimine)- $\mu$ -aquo-dicobalt in oxygen at various pressures. Temperature constant: 25–27°. Pressure in mm. of Hg.

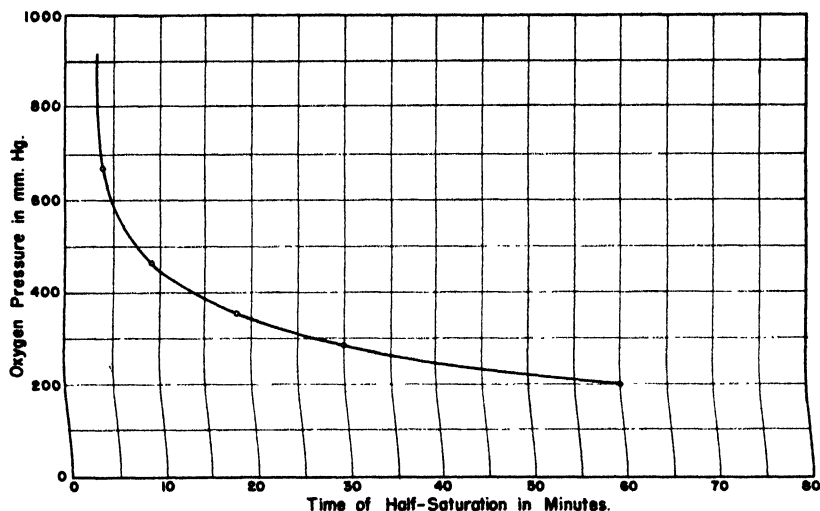


FIG. 2. Effect of pressure on the rate of oxygenation of bi-(disalicylaethylenediimine)- $\mu$ -aquo-dicobalt.

in Figures 3 and 4, the rate of oxygenation fell off at higher temperatures and again at lower temperatures. The experimental error in this series of measurements was rather high and the data could not always be exactly reproduced, owing probably to some factor related to the activation of the material. It became apparent, however, that the optimum temperature of oxygenation was close to  $20^{\circ}$  and that increasing the pressure of oxygen beyond 900 mm. of mercury could not significantly

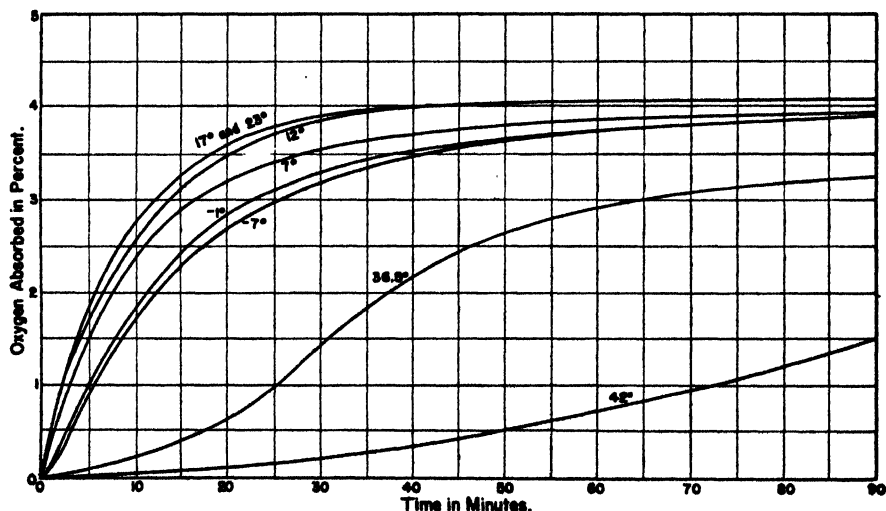


FIG. 3. Rate of oxygenation of bi-(disalicylaethylenediimine)- $\mu$ -aquo-dicobalt in oxygen at various temperatures. Pressure constant: 510 mm. Hg.

decrease the time which was required to oxygenate the compound. Judging from the curves a temperature of  $45^{\circ}$  to  $50^{\circ}$  should be ample to effect the deoxygenation of the compound at atmospheric pressure. This was found experimentally to be true.

Assuming that the oxygen pressure-oxygenation rate relationship will hold when air is used as the oxygenating agent if the air is circulated freely, an air pressure of 100 pounds should oxygenate the compound in eight to ten minutes.

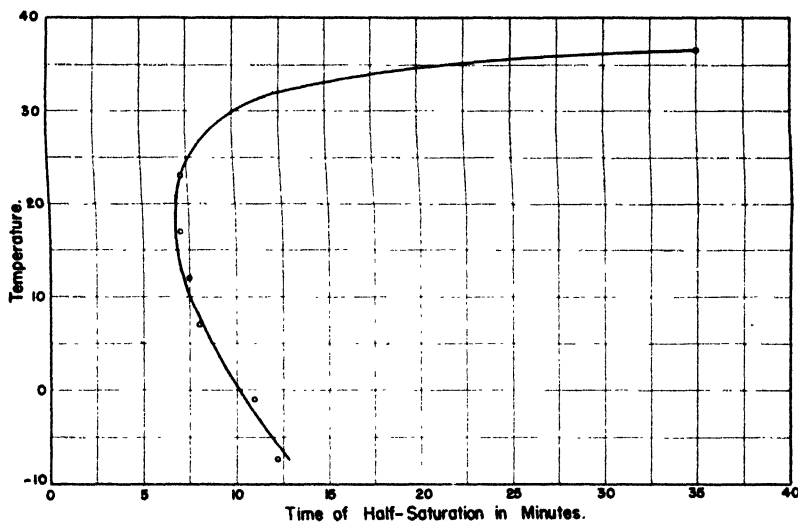


FIG. 4. Effect of temperature on the rate of oxygenation of bi-(disalicylaethylenediimine)- $\mu$ -aquo-dicobalt.

The time required for oxygenation was found to be much greater if the heat of oxygenation was retained in the material. The temperature of the material rose about  $15^{\circ}$  under adiabatic conditions.

The rate of oxygenation of bi-(disalicylaethylenediimine)- $\mu$ -aquo-dicobalt in oxygen was also determined at various temperatures with a second apparatus; see Paper XIV, Figure 1.

#### THE OLIVE BY-PRODUCT

As indicated in Paper II, Co-Ox is converted by water, particularly when hot and in the presence of acid, to an olive-colored material which does not absorb oxygen. In the absence of other obvious reasons, the preparation of a batch of material having an oxygen-carrying capacity below normal is probably due to the formation of some olive by-product.

It was found possible to convert a preparation of Co-Ox which had been only somewhat impaired by the formation of the olive by-product back to the oxygen-carrying compound by heating it in a vacuum at  $170^{\circ}$ . Thus, for example, a preparation of Co-Ox having an oxygen-carrying capacity of only 4.42 per cent was raised in capacity to 4.75 per cent

by heating for 45 minutes in a vacuum at 195°. This is not only important with respect to the initial preparation of the oxygen-carrying material but also to the regeneration of oxygen-carrying material which has become impaired in use as a result of having absorbed water. However, studies made on oxygen-carrying material, which had been put through 4,400 oxygenation-deoxygenation cycles and had deteriorated in oxygen-carrying capacity to about 20 per cent of its original value, was found to be capable of some regeneration by such a heat treatment, although the improvement found was hardly enough to be of commercial significance. Thus, heating in a vacuum for 60 minutes raised the oxygen-carrying capacity of the material from 0.9 to 1.46 per cent; further heating caused a decrease in the capacity.

#### CYCLIC OXYGENATION AND DEOXYGENATION IN SOLUTION

The iron-indigo compound of Kunz and Kress (5) was shown to carry oxygen reversibly in a pyridine solution for a few cycles. Bi-(disalicylal-ethylenediimine)- $\mu$ -aquo-dicobalt also was found to absorb and release oxygen in pyridine solution but it was only possible to go through the cycle once. When the pyridine was replaced by chloroform no enrichment of the air by oxygen was observed. In view of the fact that pyridine causes the expulsion of water from the compound and therefore breaks up the binuclear compounds, this failure to carry oxygen in solution is not surprising.

#### MISCELLANEOUS PROPERTIES OF Co-Ox

Thielert and Pfeiffer (6) reported that many ferric inner complex compounds have a catalytic effect causing the luminescence of 3-amino-phthalhydrazide in an alkaline solution containing hydrogen peroxide. Bi-(disalicylal-ethylenediimine)- $\mu$ -aquo-dicobalt has this same effect, a minute amount of the compound causing a brilliant bluish luminescence.

Bi-(disalicylal-ethylenediimine)- $\mu$ -aquo-dicobalt, in either the oxygenated condition or the deoxygenated condition, acquires an electrostatic charge with great ease. In merely pouring a small quantity of the material from a piece of paper, it becomes so highly charged that it clings to the paper with considerable tenacity. Adjacent particles of the Co-Ox repel each other. The ease with which the charge is acquired and its size appear to be rather unusual.

Co-Ox has been described variously in the preceding pages as being of a maroon or cinnamon color. More precisely it matches the R3/4 shade of the Munsell Book of Color.

#### EXPERIMENTAL WORK

##### METHOD OF ANALYSIS

The usual Kjeldahl method failed to yield consistent or correct values for nitrogen on either disalicylal-ethylenediimine or its cobalt derivative. Satisfactory results were secured by treating the sample first

with dilute sulfuric acid (1:1) and then evaporating to concentrate the sulfuric acid. The digestion with a little selenium oxychloride catalyst and the usual distillation were then carried out. Presumably direct digestion with concentrated sulfuric acid converted the nitrogen to oxides of nitrogen which escaped. Preliminary treatment with dilute acid first hydrolyzed the Schiff's base and the ethylenediamine formed then yielded ammonium sulfate on digestion.

Disalicylaethylenediimine was carefully purified by recrystallization and analyzed by this modified Kjeldahl procedure. Found: 10.35, 10.34, 10.47, 10.48, per cent nitrogen; calculated for  $C_{16}H_{10}O_2N_2$ , mol. wt. 268: 10.44 per cent nitrogen.

The obvious method of determining cobalt in these compounds, that of evaporating with sulfuric acid and weighing the cobalt sulfate produced, is unsatisfactory owing to a very serious creeping of the solution over the walls of the crucible. All of the volumetric methods in the literature for cobalt were found to be unsatisfactory except the potentiometric titration in ammoniacal citrate solution with ferricyanide (1,2). The ferricyanide method yielded excellent results on known cobalt solutions. Primary standard cobalt sulfate was prepared for reference by evaporating chloropentamminocobalt chloride with sulfuric acid and heating the resulting cobalt sulfate at  $550^\circ$  for several hours. The organic cobalt compounds were decomposed by boiling with a mixture of dilute nitric and sulfuric acids, and after complete decomposition, the cobalt solution was treated with a large amount of citric acid, made ammonical, and titrated potentiometrically with ferricyanide.

#### EFFECT OF THE PRESENCE OF NICKEL

Since any conclusions drawn from the results of analyses of Co-Ox are highly subject to error if impurities are present it became pertinent to inquire into the fate of any nickel present in the cobalt salts used in the preparation of Co-Ox. The nickel in various cobalt preparations was determined by the method of Feigl and Kapulitzas (3) in which the cobalt is converted to a cobaltcyanide complex, the excess hydrogen peroxide and cyanide and the nickel cyanide complex being destroyed by the addition of formaldehyde, and the nickel precipitated with dimethylglyoxime. Found: 0.12, 0.21 per cent nickel in "C. P." cobalt chloride. Found: 0.16, 0.22 per cent nickel in Co-Ox prepared from this cobalt chloride.

Apparently nickel is carried down about quantitatively during the formation of Co-Ox. That nickel salts alone yield a compound with disalicylaethylenediimine was quickly confirmed, the product being brown in color and very insoluble. Since nickel would not be measured with the cobalt in the ferricyanide method for cobalt it was apparent that a nickel-free compound would have to be prepared if the results for cobalt were to be of significance.



NICKEL-FREE COBALT CHLORIDE THROUGH SODIUM  
AMMONIUM COBALTINITRITE

A saturated solution of 1,200 g. of cobalt chloride was prepared and filtered. Enough acetic acid was added to make the solution about 25 per cent acetic acid. Approximately 1,000 g. of sodium nitrite was added as rapidly as possible. During the next two hours more sodium nitrite and acetic acid were added whenever the evolution of gas ceased. The solution was then allowed to stand about four hours, until the reaction was nearly completed. About 400 g. of ammonium chloride was then added. After standing overnight the yellow precipitate which had formed was filtered off and dried. This sodium ammonium cobaltinitrite was ignited to the oxide in a graphite crucible. The residue of oxide was leached with water several times and then dissolved in a little less than the theoretical amount of hydrochloric acid. The excess oxide was then filtered off, the solution evaporated down, and the cobalt chloride crystallized out. It was found that if the sodium salts were not leached out before dissolving the oxide, a double chloride of cobalt and sodium formed which was not satisfactory for our purpose. The cobalt chloride obtained was analyzed for nickel by the method of Feigl and Kapulitzas (3); no nickel was present.

## RESULTS OF ANALYSES

Careful analyses were made of several different preparations of Co-Ox for nitrogen, cobalt, carbon, and hydrogen. The results on these analyses were good and indicated that the molecular weight of the compound is higher than that of the simple compound disalicylaethylenediimine cobalt, that is 325. The values for hydrogen are disconcerting in that they fail completely to show the half molecule of water per cobalt atom known to be present. As will be seen the values for hydrogen in the Schiff's base, disalicylaethylenediimine were quite satisfactory.

## RED, INACTIVE FORM OF DISALICYLAETHYLENEDIIMINE COBALT

About 40 g. of Co-Ox, oxygen-carrying capacity 4.74 per cent, was treated with 200 ml. of 3 per cent potassium hydroxide in absolute alcohol. The mixture was boiled gently for 20 minutes. In the course of a few minutes the cinnamon-colored, oxygen-carrying compound was changed to a cherry-red, crystalline material. After cooling, the material was filtered and washed well with water, until the filtrate was colorless. The material was then pressed between layers of cloth in a hydraulic press exerting about 0.75 ton per square inch, which removed most of the water. It was then dried in a vacuum for 30 minutes at 100°. Yield: approximately quantitative; oxygen-carrying capacity: 0.02 per cent. Found: 8.30, 8.32, 8.42 per cent N by the modified Kjeldahl method; 17.75, 17.89, 17.67 per cent Co, by ferricyanide titration; calculated for  $C_{16}H_{14}O_2N_2Co$ , mol. wt.: 325: 8.61 per cent N, 18.13 per cent Co.

The cobalt-nitrogen ratio calculated from these data is almost exactly one to two but the results of both are low, the molecular weight calcu-

TABLE 1  
SUMMARY OF RESULTS OF ANALYSES OF CO-OX AND OF THE SCHIFF'S BASE,  
DISALICYLALETHYLENEDIIMINE

Preparation	For	Percentage Found	Average Percentage	Calculated Molecular Weight
Co-Ox (V-18) . . . .	N	8.43, 8.40, 8.32	8.39	334
	Co	18.02	18.02	326
	C*	57.9, 57.9, 58.1	57.97	332
	H	4.32, 4.27, 4.33, 4.30, 4.28	4.30	328
Co-Ox (V-23-A) . . .	N	8.56, 8.58, 8.59, 8.49	8.55	328
	Co	17.79, 17.76, 17.70	17.75	332
Co-Ox (V-23-B) . . .	N	8.15, 8.22, 8.24, 8.15	8.19	3.42
Schiff's Base . . . .	H	6.00, 6.00, 6.00, 6.00, 6.01, 6.12	6.02	6.00†
	C‡	69.7, 70.6, 67.0, 72.0, 71.8, 71.75	70.46	71.5

\* Oxides of nitrogen removed by sulfuric acid-chromate mixture.

† Theoretical value.

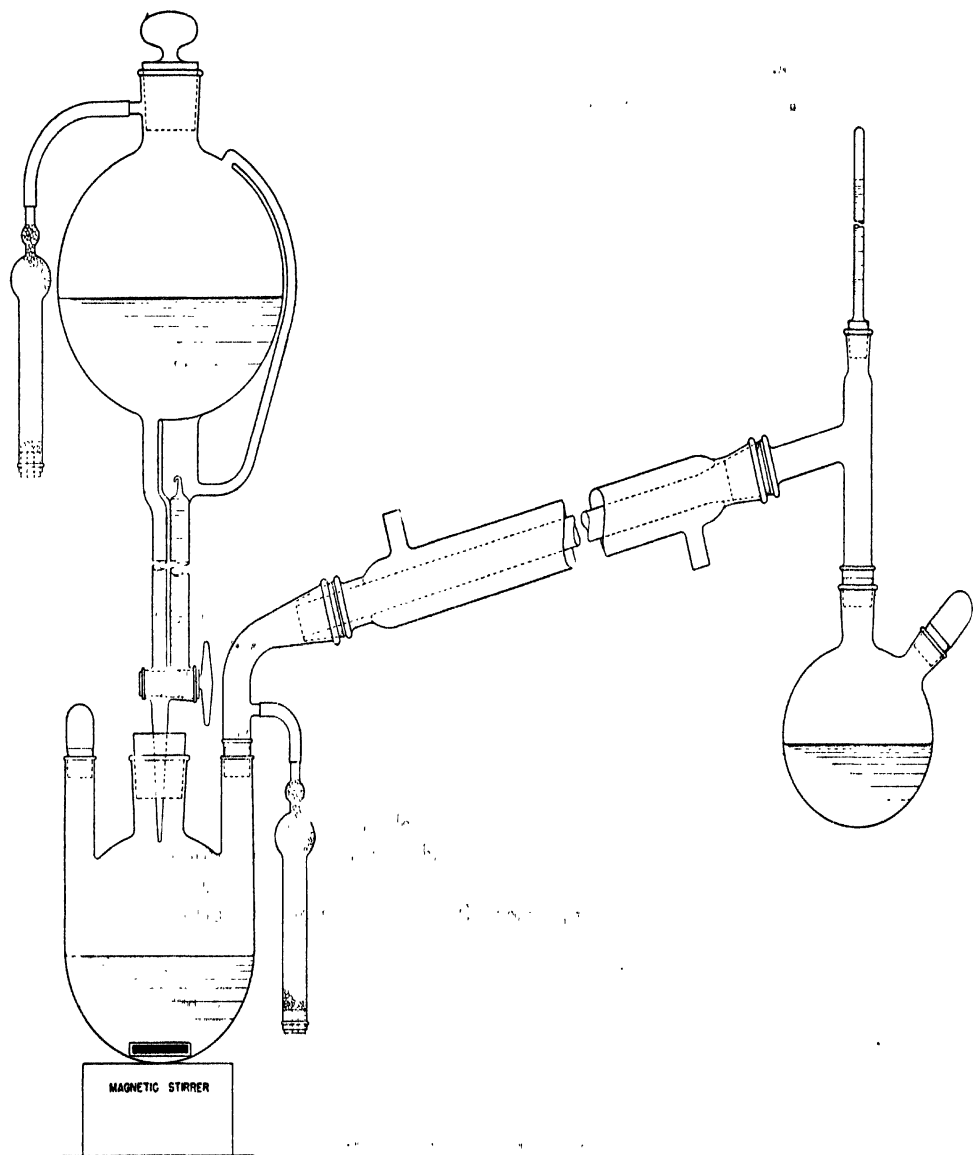
‡ Oxides of nitrogen removed with reduced copper spiral.

lated from the nitrogen and cobalt analyses are 336.0 and 333.5, respectively, indicating some impurity, possibly a half molecule of water. It is apparent that this material is isomeric with the oxygen-carrying compound, Co-Ox.

During efforts to prepare a thin layer of the bright red compound on glass in order to measure its absorption spectrum, the bright red compound was ground with mineral oil and powdered lucite in an agate mortar. Strangely, its color changed from bright red to a brown of the same shade as the oxygen-carrying compound. After this transformation, the material absorbed oxygen turning black and released oxygen returning to the cinnamon brown color. It was also found that grinding the bright red compound with mineral oil and potassium chloride produced the same effect. No quantitative data were secured as to the quantity of oxygen taken up owing to the contamination of the bright red compound with mineral oil and potassium chloride or lucite. One preparation was washed with benzene to remove the mineral oil; after this treatment the material was again bright red in color and inactive.

#### THE DIRECT DETERMINATION OF WATER IN CO-OX

The apparatus shown in Figure 5 was used. The 500 ml., two-necked flask served as a distilling flask. The sample was introduced through the neck closed by the ground glass plug which was removed only very briefly for the introduction of the sample or of pyridine. The distilling head, thermometer, condenser, adapter, and titration vessel were



**FIG. 5.** Apparatus for the direct determination of water.

all connected by ground glass joints. The drying tube attached to the adapter was filled with anhydrous calcium sulfate. The titration vessel consisted of a 1 l. three-necked flask. The Karl Fischer reagent was dispensed from a Machlett buret, the tip of which passed through a rubber stopper inserted in the central neck of the flask. The third neck was closed by a ground glass stopper. The flask was emptied by suction through a tube inserted through this neck so that it was unnecessary to disassemble the apparatus at any time. The times the flasks were open to the atmosphere were held to the very minimum necessary to pipet in the samples. The solution in the titration vessel was stirred magnetically.

The determination was carried out by placing about 100 ml. of methanol in the titration vessel and about 100 ml. of pyridine in the distilling flask. The pyridine was then distilled into the titration vessel until about 30 ml. remained in the distilling flask. The methanol-pyridine solution was then titrated with the Karl Fischer reagent. Exactly 100 ml. of anhydrous pyridine was then added to the distillation flask and distilled until about 30 ml. remained. The solution was titrated with the reagent and this volume designated as the pyridine blank. A second 100 ml. of pyridine was then distilled as a check on this blank. The weighed sample and 100 ml. of pyridine were then introduced into the distilling flask and 100 ml. of pyridine distilled over, taking about 30 minutes. The solution was then titrated, representing the water in 100 ml. of pyridine plus the water derived from the compound, the latter being obtained by subtracting the pyridine blank. To make certain the water was expelled from the compound a further 100 ml. of pyridine was added, distilled, and titrated. The small volume required to titrate this portion in excess of the pyridine blank was added to the volume of reagent used for the water derived from the compound.

The Karl Fischer reagent was standardized by titrating 50.0 ml. portions of a standard water in methanol solution made by weighing water from a weight buret. A blank on the methanol was run on methanol put through identical treatment without added water. The anhydrous methanol was prepared by distillation from magnesium turnings treated with a little mercuric chloride. The anhydrous pyridine was prepared by distillation from freshly broken walnut potassium hydroxide.

The samples of Co-Ox were dried in a vacuum at 120° prior to the analysis.

Found on sample A (prepared from the Schiff's base by Method B of Paper II; oxygen-carrying capacity: 4.64 per cent): 2.21, 2.26 per cent water, on sample B (sample A recrystallized from benzene; oxygen-carrying capacity: 4.79 per cent): 2.47 per cent water; calculated for  $C_{16}H_{14}O_2N_2 \cdot \frac{1}{2} H_2O$ : 2.69 per cent water.

#### ACTION OF CARBON MONOXIDE ON CO-OX

Carbon monoxide was generated by the action of sulfuric acid on formic acid. The carbon monoxide was scrubbed with alkali and col-

lected over water, precautions being taken to eliminate all traces of air from the retaining liquid and apparatus. A weighed sample of Co-Ox was placed in a glass tube and the tube evacuated with an oil pump. Carbon monoxide was then admitted to the tube until atmospheric pressure was attained. After 18 hours at room temperature the color of the compound was unchanged and the material had not gained in weight.

#### ACTION OF NITRIC OXIDE ON CO-OX

Nitric oxide was generated by the action of sulfuric acid on sodium nitrite, washed with sodium hydroxide, and collected over water. A sample of deoxygenated Co-Ox was placed in a glass tube at room temperature, the tube evacuated with an oil pump, and nitric oxide then admitted to the tube until the pressure reached atmospheric pressure. The compound immediately turned a dark blue in color and absorbed nitric oxide with the evolution of considerable heat. The reaction was complete within three minutes. The sample gained 9.31 per cent in weight. On heating the material for 30 minutes in a vacuum at 100°, a loss in weight of 0.18 per cent was observed; on further heating at 174° for six hours in a vacuum a further loss in weight of 3.70 per cent occurred.

One molecule of nitric oxide per molecule of Co-Ox corresponds to a gain in weight of 8.98 per cent (mol. wt. Co-Ox: 334). The observed value 9.31 per cent is thus slightly higher than the figure for one mole of nitric oxide per one cobalt atom: this might be explained by the presence of some nitrogen dioxide in the nitric oxide used in the experiment (see below).

#### ACTION OF NITRIC OXIDE ON RED, INACTIVE DISALICYLALETHYLENEDIIMINE COBALT

A weighed sample of the bright red, inactive compound was placed in a tube, the tube evacuated with an oil pump, and nitric oxide admitted until atmospheric pressure was attained. The bright red color changed slowly to a dark brown, the reaction between this compound and nitric oxide being a great deal slower than the reaction between Co-Ox and nitric oxide. After about ten hours the material was found to have gained 12.5 per cent by weight. The dark brown, nitric oxide containing compound was then heated at 170° in a vacuum for six hours; a loss in weight of 12.22 per cent was observed. It appears therefore, that the nitric oxide is reversibly absorbed by the bright red compound.

#### ACTION OF NITROGEN DIOXIDE ON CO-OX AND INACTIVE DISALICYLALETHYLENEDIIMINE COBALT

Nitrogen dioxide was prepared by oxidizing dry nitric oxide prepared by the action of sulfuric acid on potassium nitrite. Sulfuric acid was slowly dropped on a 25 per cent solution of potassium nitrite. The nitric oxide evolved was dried over phosphorous pentoxide, and then

mixed with pure, dry oxygen in an Erlenmeyer flask containing glass beads cooled in an ice-bath. The gas condensed to a green-blue liquid, a mixture of nitric oxide and nitrogen dioxide. Oxygen was bubbled through the liquid until the color changed to a light tan. The liquid was stored at  $-20^{\circ}$ . Weighed samples of disalicylaethylenediimine, of deoxygenated Co-Ox, and of inactive disalicylaethylenediimine cobalt were placed in a bottle and the bottle evacuated. Nitrogen dioxide was admitted slowly until atmospheric pressure was attained. After fifteen hours the samples were removed and weighed. All three materials had absorbed nitrogen dioxide and turned dark brown in color. Co-Ox picked up 56.1 per cent in weight, its red inactive isomer 48.7 per cent, and the Schiff's base 70 per cent. These gains correspond respectively to 3.97, 3.45, and 4.06 molecules of nitrogen dioxide per molecule of compound. It was apparent that the nitrogen dioxide attacked the organic molecule, undoubtedly at the double bonds and that the absorption was of a character entirely different from the absorption of oxygen or nitric oxide.

#### ACTION OF NITROUS OXIDE ON CO-OX

Nitrous oxide was prepared by heating a mixture of ammonium nitrate and sea sand to  $200^{\circ}$ . The gas was collected over water. Weighed samples of deoxygenated Co-Ox and its inactive, red isomer were placed in a glass tube, the tube evacuated, and nitrous oxide admitted until atmospheric pressure was attained. After ten hours the Co-Ox had gained 0.34 per cent in weight and the inactive isomer 0.18 per cent. The gains in weight absorbed were probably due to oxygen in the nitrous oxide derived from the water retaining liquid or possibly to nitrous oxide remaining mixed with the powdered materials on the boat. In any case nitrous oxide did not appear to be absorbed.

#### MAGNETIC SUSCEPTIBILITY MEASUREMENT

The magnetic susceptibility measurements were made with a Gouy type apparatus, using a field of about 12,000 gauss. A standard nickel chloride solution, 1.336 *M*, was prepared from Mond nickel and used for calibration. The volume susceptibility used was  $5.332 \times 10^{-6}$  per ml. (8). The sample tube was identical with the one described by Freed (8) being made from pyrex tubing 3 mm. in diameter with a thin uniform partition at the center. The tube was checked for magnetic symmetry at various field strengths. The usual precautions were taken to eliminate temperature effects and ferromagnetic impurities. The tube was calibrated before and after a measurement on the oxygen-carrying compound to insure that the field strength and other factors remained constant. A current of 22 amperes was used, well into the saturation region of the magnet.

The oxygen-carrying compound was prepared from cobalt sulfate prepared by decomposing chloropentamminocobalt chloride; in this way the absence of impurities of iron and nickel was guaranteed.

The following values were obtained for the molar susceptibilities:

Co-Ox, deoxygenated	$X = 316 \times 10^{-5}$ $\mu = 2.73$
Co-Ox, oxygenated	$X = 18.3 \times 10^{-5}$ $\mu = 0.656$
Red, inactive isomer	$X = 269 \times 10^{-5}$ $\mu = 2.52$

The molecular weight of 334 was used in making these calculations, that is, the molecular weight of one cobalt atom, with its associated Schiff's base and half molecule of water.

#### DENSITY OF DEOXYGENATED AND OXYGENATED CO-OX

The densities of the deoxygenated and oxygenated forms of bi-(disalicylaethylenediimine)- $\mu$ -aquo-dicobalt were determined under toluene in a Geissler specific gravity bottle. The density was calculated from the density of toluene at the temperature of the determination and from the weight of toluene displaced by the compound.

Deoxygenated form: 1.526, 1.521

Oxygenated form: 1.618, 1.605

#### CYCLIC OXYGENATION AND DEOXYGENATION IN SOLUTION

The apparatus employed is pictured in Figure 6. Provision was made for saturating the solution containing the Co-Ox with air, and for then

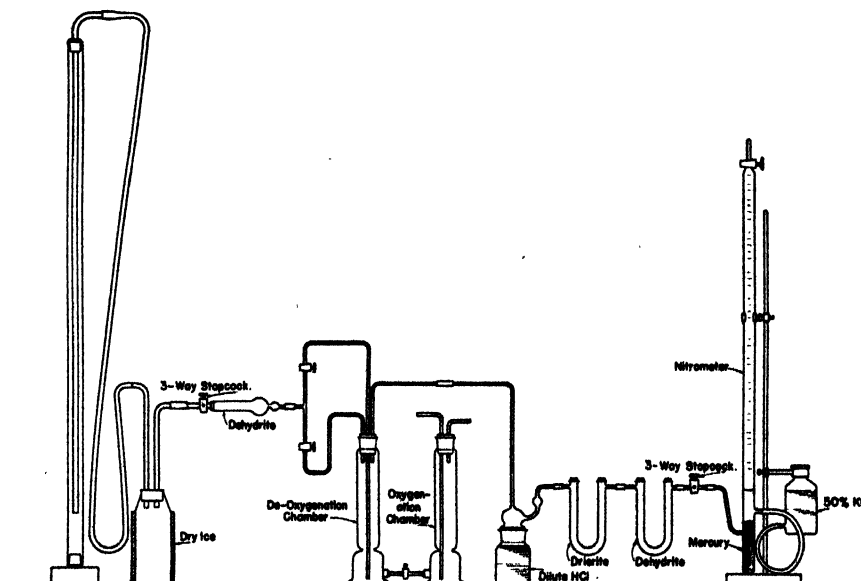


FIG. 6. Apparatus for testing cyclic operation of bi-(disalicylaethylenediimine)- $\mu$ -aquo-dicobalt in solution.

transferring the solution to another vessel in which the air was swept out of the solution with a stream of carbon dioxide gas. The gaseous mixture was then passed through a potassium hydroxide solution in a nitrometer where the carbon dioxide was absorbed and the remaining oxygen collected. By carrying out the oxygenation and deoxygenation steps in separate vessels, the danger of admitting air during the deoxygenation step was avoided. Provision was made for absorbing the solvent swept from the deoxygenation vessel by the carbon dioxide stream. The nitrometer used for measuring the evolved gases was filled with a 50 per cent solution of potassium hydroxide.

The procedure adopted in carrying out the oxygenation-deoxygenation cycle on a sample was as follows: A quantity of 240 ml. of pyridine, b.p. 113.5–114.5°/740 mm., was introduced into the oxygenation chamber. The apparatus was swept out with carbon dioxide until only microbubbles were observed to reach the top of the nitrometer. The sample of pyridine was then transferred into the deoxygenation chamber and the carbon dioxide bubbled through the pyridine until microbubbles were again obtained. This process required about 45 minutes. It was now assumed that the pyridine was freed of all previously absorbed gases.

A blank determination on the pure pyridine was made by transferring the solution to the oxygenation chamber and drawing air through the solution by means of a water aspirator for a definite period of time, usually five or ten minutes. The solution was again returned to the deoxygenation system and carbon dioxide bubbled through until all absorbed air was removed as shown by the size of the bubbles reaching the top of the nitrometer.

The pyridine was transferred again to the oxygenation chamber to which had been added a weighed sample of deoxygenated Co-Ox. Air was drawn through the pyridine solution by means of a water aspirator for the same period of time as in the blank run. The pyridine solution was transferred again to the deoxygenation chamber and carbon dioxide bubbled through the pyridine solution until all absorbed air was removed as shown by the microbubbles reaching the top of the nitrometer.

In some of the preliminary work oxygen gas was used in place of air. The gas obtained from the pyridine solution was rich in oxygen as demonstrated by the fact that a glowing splint was immediately ignited when placed in an atmosphere of the gas.

It seemed probable that some pyridine vapor was not absorbed by the sulfamic acid and anhydrous used in the purification train, and, therefore, was present in the gas finally collected in the nitrometer. This conclusion was drawn from the fact that in two instances when testing for oxygen with a glowing splint an explosion resulted inside the nitrometer. For the subsequent work the purification train described in the drawing was replaced by a gas wash bottle containing 1 N hydrochloric acid and followed by a U-tube containing anhydrous calcium sulfate and another containing anhydrous magnesium perchlorate. The gas collected was analyzed for oxygen. Results are shown in Table 2.



TABLE 2  
SUMMARY OF RESULTS USING PYRIDINE  
Volume of pyridine: 240 ml.

Experiment Number	Time of Saturation, Minutes	Gas Used	Weight of Sample, Grams	Volume Collected, Ml.	Oxygen Content, Percentage
1-A.....	10	O <sub>2</sub>	blank	17.5	none, ignited glowing splint
1-B.....	10	O <sub>2</sub>	0.8369	18.2	
2-A.....	10	O <sub>2</sub>	blank	28.2	81
2-B.....	10	O <sub>2</sub>	0.9660	28.1	86
3-A.....	10	air	blank	15.5	21
3-B.....	10	air	0.949	22.5	45
3-C.....	10	air	0.949	14.8	21
4-A.....	15	air	blank	14.2	21
4-B.....	15	air	0.979	22.5	57
4-C.....	5	air	0.979	14.2	20

The blank obtained was apparently the result of the absorption of air by pyridine since the oxygen content of the gas collected was about the same as that of air. Upon the introduction of the oxygen-carrying compound an additional amount of gas was obtained, which upon analysis was shown to contain more oxygen. Evidently the cycle cannot be repeated as the gas collected upon repeating the oxygenation-deoxygenation cycle had the same oxygen content as the blank.

The procedure was repeated using chloroform as the solvent. The drying agents following the deoxygenation chamber were replaced with a dry ice trap. These results are summarized in Table 3.

TABLE 3  
SUMMARY OF RESULTS USING CHLOROFORM  
Volume of chloroform: 240 ml.

Experiment Number	Time of Saturation, Minutes	Gas Used	Weight of Sample, Grams	Volume Collected, Ml.	Oxygen Content, Percentage
1-A.....	10	air	blank	27	29
1-B.....	10	air	0.5810	27	28.5
2-A.....	10	air	blank	28	26.8
2-B.....	10	air	0.4630	32	25

The results indicate that no increase in the oxygen content of the gas absorbed resulted when the compound was dissolved in chloroform. Evidently the chloroform itself has a preferential solubility for oxygen since even the blanks showed a higher percentage of oxygen than in air.

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[Further discussions of the "Studies on Oxygen-Carrying Cobalt Compounds" will follow in subsequent issues of the *Journal of Science*.]



# STUDIES ON OXYGEN-CARRYING COBALT COMPOUNDS<sup>1</sup>

## IV. THE REACTION OF DISALICYLALETHYLENEDIIMINE AND COBALT CHLORIDE UNDER ANHYDROUS CONDITIONS

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It was observed that when the oxygen-carrying compound, bi-(disalicylalethylenediimine)- $\mu$ -aquo-dicobalt, was prepared by the addition of an aqueous, alkaline solution of the Schiff's base, disalicylalethylenediimine, to an aqueous solution of a cobalt salt, an orange precipitate formed first which was quickly converted to the cinnamon-colored, oxygen-carrying compound or covered up by it. The formation of this orange precursor was not observed when the oxygen-carrying compound was prepared by direct interaction of the cobalt salt, the ethylenediamine, and the salicylaldehyde or in the pyridine method of preparing the material. The nature of this orange precursor was naturally of considerable interest in that it could possibly yield some clue to the unique character of the oxygen-carrying compound.

Inasmuch as the orange material existed only momentarily in water solution, it was fruitless to attempt its preparation in the presence of water. The method of attack, therefore, was to work under anhydrous conditions, preventing as much as possible the contact of the compound with moist air. Anhydrous ethyl alcohol was selected as the medium.

As expected the orange material was obtained but in addition a green and a purple compound were also obtained. By careful fractional crystallization it was possible to obtain a fairly pure specimen of the orange compound and a fair specimen of the green compound but the purple compound appeared to be rather badly contaminated with other material.

The orange compound was inactive toward oxygen and not hygroscopic. On treatment with water it promptly passed into the cinnamon-colored, oxygen-carrying compound. It contained chloride. The chemical analysis gave the ratio  $\text{Co} : \text{N} : \text{Cl} :: 3 : 4 : 2$ . This indicated the composition to be  $(\text{C}_{10}\text{H}_{14}\text{O}_2\text{N}_2\text{Co})_2 \cdot \text{CoCl}_2$ , which is possibly a binuclear compound in which two cobalt atoms are joined through two bridging chloro groups, that is, cobalt bi-(disalicylalethylenediimine)- $\mu$ -dichlorodicalatoate. Such bridging chloro groups are unknown in the coordination chemistry of cobalt but are common among the palladium and platinum compounds.

On treatment with water cobalt chloride was released from the

<sup>1</sup> Papers I, II, and III of this discussion were published in this Journal in Vol. XXI, pp. 271-309, 1947.

orange compound and two molecules of water entered, one of which was expelled at 100° to yield the oxygen-carrying compound.

The green compound crystallized from the filtrate from the orange material on evaporation. Inasmuch as three cobalt atoms but only two molecules of disalicylaethylenediimine entered the orange compound, the solution from which the green material crystallized contained the Schiff's base in excess of the cobalt. It contained also the hydrochloric acid liberated in the formation of the orange compound. The green compound contained chloride. Analysis for cobalt, nitrogen, and chloride indicated a ratio of Co : N : Cl :: 1 : 3.5 : 4.5. It is probable that the compound contained more than one molecule of disalicylaethylenediimine per cobalt atom but its exact nature can only be decided on the basis of further work.

On treatment with water the green compound was converted to the oxygen-carrying compound.

The nature of the purple compound is even more obscure. It was obtained only in small amounts and in very impure form. It did not yield the oxygen-carrying compound on treatment with water.

Although the nature of the green and purple compounds has not been elucidated, the composition of the orange compound is certain and the work establishes beyond a doubt that water is essential to the formation of and present in the oxygen-carrying compound.

## EXPERIMENTAL WORK

### THE REACTION OF DISALICYLAETHYLENEDIIMINE AND COBALT CHLORIDE UNDER ANHYDROUS CONDITIONS

To a solution of 13.4 g. of disalicylaethylenediimine in 500 ml. of hot alcohol was added a solution of 6.3 g. of anhydrous cobalt chloride dissolved in 100 ml. of hot, absolute alcohol. The solution was boiled for a minute while scratching the beaker with a stirring rod. An orange precipitate appeared identical in color with the orange precursor observed in the preparation of the oxygen-carrying compound. This material was filtered off while the solution was still hot, washed with absolute alcohol, and dried at 100° in a vacuum. Yield: 3 g.

The filtrate was heated gently on a hot plate for several minutes. A green, crystalline material appeared causing severe bumping. This material was filtered off and dried. Yield: 3 g.

The filtrate from the green material was boiled gently for about ten minutes at which time it began to bump severely. Filtration yielded 2 g. of dark purple crystals.

Further evaporation of the filtrate gave a mixture of the green and purple compounds. Yield: 1.5 g.

This reaction and the fractional crystallization were repeated a number of times. Prolonged washing of the orange material with absolute alcohol did not remove all of the chloride from the compound. The fractionation was not as clean cut as could be desired, the orange material always being contaminated by some of the green compound.

One preparation was made in a modified Zerwitinoff bulb with a fritted glass filter attached by a ground glass joint so that the entire operation could be conducted in the absence of air. The apparatus was flushed out with nitrogen, the two solutions were boiled to expel air, and the solutions brought together and the orange precipitate filtered off and washed without exposure to air. Both the orange and the green compounds were obtained and the possibility is thus excluded that the green compound is an oxidized, trivalent cobalt compound.

The reaction was also run a number of times using the disodium and dipotassium salts of disalicylalethylenediimine rather than the Schiff's base itself. The salts were obtained by treating alcohol solutions of the Schiff's base with the requisite amounts of sodium or potassium hydroxide and allowing the solution to cool and crystallize. The materials obtained were mixtures of the free Schiff's base and its salts owing to hydrolysis. When used subsequently the fractionation of the orange, green, and purple compounds was complicated by the presence of insoluble sodium chloride, potassium carbonate, and tarry materials of unknown composition. The procedure was thus much less satisfactory than the one described above using the Schiff's base.

#### THE ORANGE COMPOUND

Attempts to recrystallize the orange compound from cellosolve, butyl cellosolve, and carbitol led to greenish-black solutions from which neither the green nor the orange compound could be crystallized. The orange compound, after having been dried at 100° in a vacuum, was placed in an atmosphere of oxygen at 200 pounds pressure. It did not gain in weight or change in color and thus was shown to be inactive toward oxygen. Nor was it hygroscopic.

On treatment with water the orange material was converted immediately to a cinnamon-colored material capable of repetitively absorbing and releasing oxygen. Undoubtedly this material was identical with the original oxygen-carrying compound. The treatment was carried out somewhat more carefully by treating a sample of the orange material, weighing 0.5113 g., on a Buchner funnel fitted with a double thickness of filter paper, with boiling water. The material changed rapidly in color to a cinnamon brown and considerable material passed into the filtrate. The residue weighed 0.3745 g., a loss in weight of about 26 per cent. This is a considerably greater loss than would have been expected knowing the approximate solubility of the oxygen-carrying compound. In another experiment a sample of the orange compound weighing 0.3529 g. was treated with hot water on a boat, and the water evaporated away. The residue was dried in a vacuum at 100° for thirty minutes and was found to have gained 3.06 per cent in weight and to then carry 3.14 per cent oxygen. After a further period of drying at 100°, the gain in weight was reduced to 2.64 per cent and the oxygen-carrying capacity had increased to 3.31 per cent. Thus, in this case the compound did not dehydrate as readily as the material obtained when the orange material was treated on the funnel and a part of the material washed into the filtrate.

The orange compound when treated with dilute alkali passed directly into the bright red, inactive isomer of the oxygen-carrying compound (see Paper III of this series).

The nitrogen in the orange material was determined by our modified Kjeldahl method, that is, the digestion was begun with dilute sulfuric acid (1 : 1). Found: 6.78, 6.75 per cent N.

The cobalt was determined by decomposing a weighed sample by digestion with sulfuric acid and nitric acid, expulsion of the nitric acid, and potentiometric titration with ferricyanide in ammoniacal, citrate solution. Found: 20.92, 21.01, 20.92 per cent Co.

The chloride in the material was determined by the Thompson-Oakdale method (1). A blank was run and applied. Found: 9.14, 8.65, 9.14 per cent Cl.

These results give an empirical formula of  $\text{Co}_{0.354}\text{N}_{0.516}\text{Cl}_{0.258}$  or ratios of  $\text{Co} : \text{N} : \text{Cl} = 3 : 4.37 : 2.18$ . Owing to some contamination of the orange compound by the green compound the cobalt analysis is probably low and the nitrogen analysis high (see below for the composition of the green compound) so that the most reasonable ratio is  $\text{Co} : \text{N} : \text{Cl} = 3 : 4 : 2$ , corresponding to the formula  $(\text{C}_{16}\text{H}_{14}\text{O}_2\text{N}_2\text{Co})_2 \cdot \text{CoCl}_2$ , mol. wt.: 780, containing 22.6 per cent Co, 7.17 per cent N, and 9.10 per cent Cl.

#### THE GREEN COMPOUND

When placed in an atmosphere of oxygen at 200 pounds pressure the green compound did not gain in weight or change in color and was thus shown to be inactive toward oxygen. On treatment with water the green compound was rapidly converted to the cinnamon-colored, oxygen-carrying compound. This was carried out on a filter and the residue washed with water and dried in a vacuum at  $100^\circ$ . The oxygen-carrying capacity of the product was 3.3 per cent.

It was suspected that hydrochloric acid was involved in the formation of the green compound, the green compound possibly being a hydrochloride of the orange. A pellet of the orange compound when dropped into concentrated hydrochloric acid immediately turned green, changing to the color of a cobalt salt in hydrochloric acid only after a period of fifteen minutes. A sample of the orange material was placed in an atmosphere of hydrogen chloride generated in the bottom of a large beaker by the action of hydrochloric acid on sulfuric acid. In about three hours the mass had turned green, matching the color of the green compound.

The green compound was analyzed for cobalt and nitrogen by the methods mentioned above. Found: 14.18, 14.09, 14.22 per cent Co; 12.40, 12.34 per cent N. The chloride in the compound was determined by treating a mixture of the weighed sample with an excess of silver nitrate, warming gently, then diluting with a little water, and digesting until clear. This was carried out in a large, conical flask the mouth of which was closed with a funnel. The solution was diluted to 300 ml., boiled for a few

minutes, and the determination completed gravimetrically. Found: 38.99, 38.58 per cent Cl.

These analyses give the ratios: Co : N : Cl :: 1 : 3.48 : 4.57. It is difficult to assign a likely formula and constitution to such an empirical formula.

#### THE PURPLE COMPOUND

The final stages of the fractional crystallization yielded a purple compound which, like the orange and green compounds, was inactive toward oxygen but did not yield a material active toward oxygen on treatment with water. It was obviously not very pure.

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## STUDIES ON OXYGEN-CARRYING COBALT COMPOUNDS

### V. DI-(2-HYDROXY-3-NITROBENZAL)-ETHYLENEDIIMINE COBALT. 3-NITRO CO-OX

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Of the very limited number of substituted salicylaldehydes which yielded ethylenediamine-cobalt compounds reversibly active toward oxygen, 2-hydroxy-3-nitrobenzaldehyde (3-nitrosalicylaldehyde) was found to be especially interesting in that it yielded a cobalt compound which absorbed oxygen more rapidly and which possessed certain chemical characteristics absent in Co-Ox but characteristic of this class of oxygen-carriers in general.

Salicylaldehyde was nitrated directly in acetic acid with fuming nitric acid, which leads to a mixture of the 3-nitro- and 5-nitro- isomers. Since a considerable quantity of 2-hydroxy-3-nitrobenzaldehyde was needed, the nitration reaction and the subsequent separation of the 3- and 5-nitro- isomers were studied in detail and the best conditions established for the processes. Yields of the combined isomers of 90 per cent were secured. The separation was accomplished by the fractional crystallization of the sodium salts from water.

Although 2-hydroxy-5-nitrobenzaldehyde was obtained in pure form, 2-hydroxy-3-nitrobenzaldehyde was obtained in two crystal forms, possibly hydrogen bonding isomers. The chemical identity of the two forms was established and it was found that only the high melting form, obtained from alcohol as large brown crystals, was capable of producing an ethylenediamine-cobalt compound active toward oxygen.

The Schiff's base, di-(2-hydroxy-3-nitrobenzal)-ethylenediimine, was prepared by mixing equivalent amounts of 2-hydroxy-3-nitrobenzaldehyde and ethylenediamine in a warm alcohol solution. From the high-melting form of 2-hydroxy-3-nitrobenzaldehyde a yellow condensation product was obtained containing water of crystallization. On drying or heating, this yellow material was dehydrated to an orange compound which readily rehydrated on exposure to moisture. Recrystallization of this material from a high boiling solvent yielded a dark red compound isomeric with di-(2-hydroxy-3-nitrobenzal)-ethylenediimine. When the low-melting form of 2-hydroxy-3-nitrobenzaldehyde was used, other forms of di-(2-hydroxy-3-nitrobenzal)-ethylenediimine were obtained, pink in color, which subsequently yielded cobalt compounds inactive toward oxygen.

Di-(2-hydroxy-3-nitrobenzal)-ethylenediimine cobalt was synthesized by several methods and a successful method of preparing a satisfactory oxygen-carrying material was evolved. Unlike the procedure for the

parent oxygen-carrying compound, it was necessary to synthesize this oxygen-carrier by the action of an aqueous solution of a cobalt salt on the solid Schiff's base since the latter was insoluble in both water and alcohol. When prepared in this manner di-(2-hydroxy-3-nitrobenzal)-ethylenediimine cobalt was obtained as a tan-colored hydrate, inactive toward oxygen. One molecule of water per cobalt atom was expelled on heating at 120° and the red material left then absorbed one molecule of oxygen (O<sub>2</sub>) per two cobalt atoms, 3.83 per cent by weight of oxygen. The best conditions for effecting this activation (dehydration) were determined.

3-Nitro Co-Ox is very hygroscopic and hydration and oxygenation go on as competing reactions when the material is exposed to moist air or oxygen. The rates at which oxygen is absorbed under various conditions of temperature, pressure, and humidity were measured. The rate of oxygenation is more or less independent of the temperature and is affected somewhat by the moisture content of the air. When the moisture content of the air is above one milligram of water per liter the competing absorption of oxygen and moisture becomes apparent, and the amount of oxygen absorbed decreases as the moisture content of the air increases.

The rate of oxygenation of 3-Nitro Co-Ox is not greatly affected by temperature. The temperature range between oxygenation and deoxygenation is rather small. The oxygenation-deoxygenation process is quite sensitive to pressure variation and accordingly the 3-nitro compound might be satisfactory for adiabatic operation (see Paper XIV).

Di-(2-hydroxy-5-nitrobenzal)-ethylenediimine cobalt did not absorb oxygen (see Paper VIII).

## EXPERIMENTAL WORK

### NITRATION OF SALICYLALDEHYDE

Salicylaldehyde was nitrated by the method of Mazzara (1). Four 500 g. batches of salicylaldehyde were nitrated in glacial acetic acid with different concentrations of nitric acid: 70, 80, 90, and 98 per cent nitric acid. Somewhat better yields of combined 3- and 5-nitro isomers were obtained using 98 per cent acid but the principal advantage of using the stronger acid was found to be the smoothness with which the reaction progressed, the reaction commencing at considerably lower temperature. In another series of nitrations the amount of nitric acid used was varied from the theoretical amount to a 150 per cent excess. The optimum amount was found to be a 50 per cent excess. In another series the amount of acetic acid used as solvent was varied from three times to seven times the amount of salicylaldehyde nitrated. The optimum was found to be about four times the volume of salicylaldehyde. As a result of these studies the following procedure is recommended:

In a 5 l., three-neck flask, equipped with a motor-driven stirrer, a dropping funnel, a thermometer, and a vacuum line to carry off the fumes, place 2,000 g. of glacial acetic acid and 500 g. of salicylaldehyde.

Cool this solution in an ice bath to 25° and then start the slow addition of the nitric acid. During the next two and one-half hours add 400 g. of 98 per cent nitric acid, sp. gr. 1.50. This acid must be added slowly and after the first 100 g. has been added the temperature should be reduced to below 15° and held below 15° until the addition is complete. After all of the acid has been added, remove the solution from the ice bath and allow it to warm to about 45°. This will take from one to two hours. When the temperature reaches 45°, immediately dilute the material by pouring it into 10 l. of water containing 1 l. of cracked ice. Let the mixture stand at least five hours, then filter and dry. The yield of mixed aldehydes from this procedure should be about 90 per cent.

SEPARATION OF 2-HYDROXY-3-NITROBENZALDEHYDE FROM  
2-HYDROXY-5-NITROBENZALDEHYDE

The 3-nitro and 5-nitro isomers were separated by the method of Miller (2). To 3 l. of water were added 400 g. of the mixture of 2-hydroxy-3-nitro- and 2-hydroxy-5-nitrobenzaldehydes and 100 g. of sodium hydroxide. The mixture was heated until everything had dissolved and the solution was allowed to cool slowly by standing overnight. The crystalline sodium 2-hydroxy-5-nitrobenzaldehyde was filtered off. The filtrate was evaporated to approximately one-third its volume and cooled. The sodium salt of 2-hydroxy-3-nitrobenzaldehyde then crystallized out. The sodium salts of the 3- and 5-nitro isomers were recrystallized from water. Both were dissolved in hot water and the solutions acidified with hydrochloric acid. The free nitro compounds precipitated out. Each was recrystallized from alcohol. M.p. 2-hydroxy-5-nitrobenzaldehyde: 123–125°; reported by Miller (2): 126°. M.p. 2-hydroxy-3-nitrobenzaldehyde: 85–95°; reported by Miller (2): 109–110°. Repeated recrystallization from alcohol had little effect in improving the melting point of the 2-hydroxy-3-nitrobenzaldehyde obtained above. The material was recrystallized also from dilute acetic acid (1:3) but the material still melted over a broad range, 85–95°. A quantity of the aldehyde was sublimed at 110° at atmospheric pressure; beautiful white needles together with fine, white powder were obtained. The needles melted sharply at 107°, the powder melted slowly over the range 85–100°. When ground up the sublimate melted over the broad range 85–100°.

The 2-hydroxy-3-nitrobenzaldehyde was distilled under vacuum, the entire material distilling at 135–140°/9 mm. The distillate immediately crystallized to a pale yellow solid. When the mass was crushed, clear, shiny crystals were found imbedded in a mass of finely crystalline, friable material. These crystals when hand-picked from the mixture were found to have a melting point of 105°. When the solid was ground, it melted slowly over the range 85–109°.

The 2-hydroxy-3-nitrobenzaldehyde was then subjected to steam distillation. The quantity of aldehyde carried over with the steam was rather small, 1.5 g. per liter of distillate. The distilled 2-hydroxy-3-nitro-

benzaldehyde crystallized in the distillate as a fine, nearly white powder, m.p.: 85–105°.

It was concluded that the low-melting material was not an oxidation product of 2-hydroxy-3-nitrobenzaldehyde but 2-hydroxy-3-nitrobenzaldehyde itself. Sublimation, sublimation in oxygen, sublimation in vacuum, steam distillation, and rapid recrystallization from alcohol did not effect a purification. Powdered material appeared to always have a low melting point. The high and low melting forms are apparently two crystal modifications of 2-hydroxy-3-nitrobenzaldehyde, possibly hydrogen bonding isomers. In any case it was believed that the solubility of the two forms should differ and because of the low energy involved in the interconversion of one to the other that the least soluble one should be formed almost exclusively if the crystallization were sufficiently slowly carried out.

A solution of 2-hydroxy-3-nitrobenzaldehyde in boiling alcohol was prepared. Water was added to the warm alcohol solution until precipitation of the 2-hydroxy-3-nitrobenzaldehyde just began. The mixture was then heated to boiling and the small amount of precipitate redissolved. The solution was then transferred to a thermos bottle and allowed to stand until cool. Approximately twenty-four hours was required for the solution to become cool during which time 2-hydroxy-3-nitrobenzaldehyde crystallized in the form of large, tan crystals. These crystals, when taken individually or when ground, had a melting point of 108.5–109.9°.

The material purified in this manner was satisfactory for subsequent work as described below.

#### DI-(2-HYDROXY-3-NITROBENZAL)-ETHYLENEDIIMINE

This Schiff's base was obtained on treating an alcohol solution of 2-hydroxy-3-nitrobenzaldehyde with an equivalent amount of ethylenediamine. Depending on the conditions, a yellow or orange precipitate was obtained. The yellow material passed over to the orange form when dried or heated to 100°. When the orange form was placed in a moist atmosphere at room temperature, it passed over to the yellow form.

Using 2-hydroxy-3-nitrobenzaldehyde having a low melting point, 80–105°, Schiff's bases were obtained which were pink to yellow in color, which did not give the orange, anhydrous form mentioned above on drying, and which did not yield satisfactory oxygen-carrying cobalt compounds.

Recrystallization of this Schiff's base from butyl cellosolve, the only solvent found in which it is appreciably soluble, gave a very deep red, finely crystalline material.

#### DI-(2-HYDROXY-3-NITROBENZAL)-ETHYLENEDIIMINE COBALT

The conventional methods of preparing cobalt derivatives of the Schiff's bases of ethylenediamine were found to be inapplicable to the preparation of di-(2-hydroxy-3-nitrobenzal)-ethylenediimine cobalt owing to the insolubility of di-(2-hydroxy-3-nitrobenzal)-ethylenediimine or its

sodium salt in all of the common solvents. However, the reaction of a solution of a cobalt salt with an aqueous suspension of di-(2-hydroxy-3-nitrobenzal)-ethylenediimine took place completely, if slowly, and a satisfactory cobalt derivative was prepared. The procedure recommended for the preparation of di-(2-hydroxy-3-nitrobenzal)-ethylenediimine cobalt follows.

Dissolve 2 moles of 2-hydroxy-3-nitrobenzaldehyde (m.p.: 108–110°) in 1.5 l. of hot 95 per cent alcohol. Without delay add to this solution 1 mole of ethylenediamine. Filter off the orange-yellow precipitate. Mix the Schiff's base without drying into 10 l. of hot water. Add 2 moles of sodium hydroxide and 2 moles of sodium acetate dissolved in 1 l. of water. To the resulting mixture add 2 moles of cobalt chloride,  $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ , dissolved in 1 l. of water. Digest the mixture on a steam bath for six to eight hours. A fine, tan precipitate forms as the reaction progresses. Filter and dry the material at 100°. Activate the dried compound either in a vacuum oven at 120° or under infra-red lamps. The material turns brick red in color and is then active toward oxygen. Theoretical oxygen capacity: 3.86 per cent.

Cobalt acetate may be used in place of the cobalt salt and sodium acetate used above.

The excess cobalt in the filtrate from the above preparation may be recovered by the addition of sodium carbonate and subsequent filtration and ignition of the precipitate to the oxide.

It was concluded from the results of numerous preparations using 2-hydroxy-3-nitrobenzaldehyde melting over the range 85–107° that the lower the melting point of the aldehyde, the lower the oxygen-carrying capacity of the cobalt derivative:

<i>M.p. of 2-Hydroxy-3-nitrobenzaldehyde</i>	<i>Oxygen-Carrying Capacity of Cobalt Derivative</i>
109–110°	3.83 per cent
108–110°	3.70
106–110°	3.62
105–109°	3.52
105–108°	3.36
106–108°	3.36
103–108°	2.7
85–95°	2.0

Theoretical capacity: 3.83 per cent

#### ACTIVATION OF 3-NITRO CO-OX

Di-(2-hydroxy-3-nitrobenzal)-ethylenediimine cobalt is first obtained in the form of a hydrate which is inactive toward oxygen. The loss of water upon drying is gradual and there seems to be no definite temperature at which the hydrate begins to decompose. The compound may be activated in air at a temperature of 125–130°. At this temperature no apparent decomposition results but the rate of activation is not rapid,

about twenty-four hours being required. Under a vacuum the activation may be carried out at a somewhat lower temperature although only very slowly at 90–95°. At 120–125° under a high vacuum the rate of activation is rapid. The material can even be activated at room temperature in a vacuum desiccator over phosphorus pentoxide; this requires several days, however, and the oxygen-carrying capacity only reaches 2 per cent.

THE RATE OF OXYGENATION OF DI-(2-HYDROXY-3-NITROBENZAL)-  
ETHYLENEDIIMINE COBALT

A number of the earlier determinations of the rate of oxygenation of di-(2-hydroxy-3-nitrobenzal)-ethylenediimine cobalt were made by measuring the increase in weight of a glass stoppered U-tube containing the compound on the passage of air or oxygen through the tube. This method was quite satisfactory, although the compound often plugged the glass wool filters and thus prevented adequate gas flow through the U-tube. The oxygenation rates were determined at various temperatures by placing the U-tube in a water bath at the temperature desired. The glass stoppered U-tubes used in these rate determinations had a diameter of about 1 cm. The temperature of the compound was probably not maintained very constant during these determinations since no provision was made for controlling the temperature of the gas moving through the compound and since the temperature of the bath was often quite removed from room temperature. This method was also very time consuming.

It was found that di-(2-hydroxy-3-nitrobenzal)-ethylenediimine cobalt absorbed water as well as oxygen if the air or oxygen used was not thoroughly dried. Therefore a study was made to determine the correlation between the quantity of water present in the gas and the rate of oxygenation, the oxygen pressure, and temperature being held constant. The humidity of the in-going gas was adjusted by passing the gas through sulfuric acid solutions of various concentrations or through suitable saturated salt solutions. The gain in weight of the U-tube was measured and the oxygen evolved and its volume determined. The weight of oxygen absorbed was then calculated and this weight of oxygen subtracted from the gain in weight of the U-tube. The difference in weight was then equal to the weight of water absorbed. The rate of absorption of water was assumed to be linear during the period of oxygenation, the rate of absorption of water per minute was calculated, and this value was applied as a correction to the weight of the U-tube at each interval during the oxygenation. There was thus obtained the rate of oxygenation plus hydration and the rate of oxygenation alone. Although this method may not be absolutely correct since the absorption of water may not have been linear, it is felt that any error involved could only be very small.

The data obtained for the rate of oxygenation plus hydration and the rate of oxygenation alone at atmospheric pressure and room tempera-

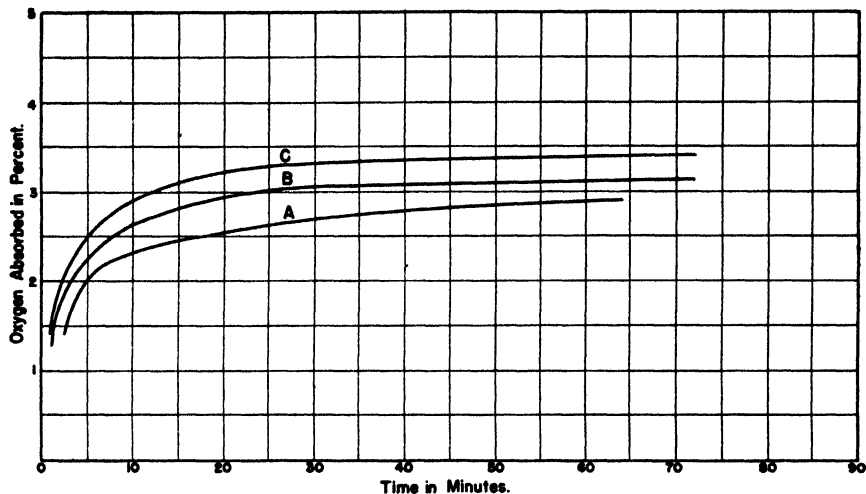


FIG. 1. Rate of oxygenation of 3-Nitro Co-Ox. A, Dry oxygen ( $\text{Mg}(\text{ClO}_4)_2$ ),  $25^\circ$ , 688 mm. Hg.; B, oxygenation; and C, oxygenation plus hydration at 5 per cent relative humidity,  $24^\circ$ , 695 mm. Hg.

ture at relative humidities of 0, 5, 10, and 56 per cent are shown in Figures 1, 2, and 3. A saturated zinc chloride solution was used to adjust the humidity of the oxygen to 10 per cent; sulfuric acid of sp. gr. 1.67 was used to adjust the humidity of the oxygen to 5 per cent; the atmosphere as used directly had a humidity of 56 per cent.

The gravimetric method, described above, for determining the rate of oxygenation of the compounds under study was rather tedious to carry out and subject to certain disadvantages. The gas volumetric method,

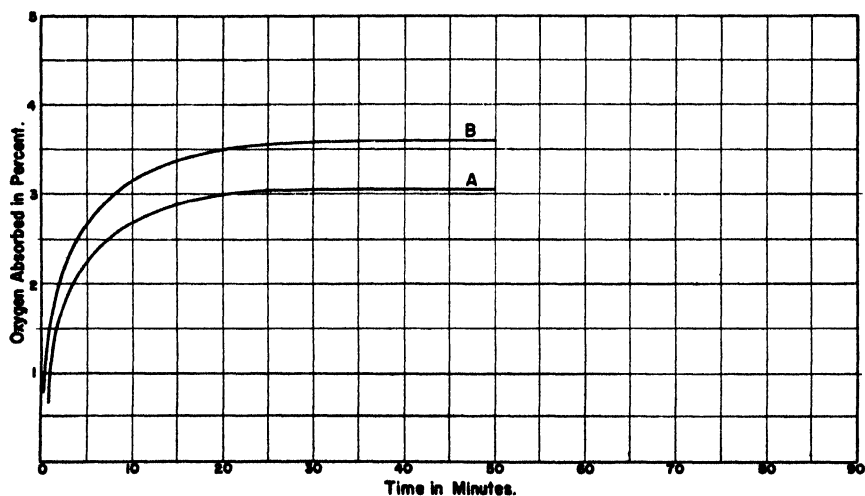


FIG. 2. Rate of oxygenation of 3-Nitro Co-Ox. A, oxygenation, and B, oxygenation plus hydration at 10 per cent relative humidity,  $26^\circ$ , 691 mm. Hg.

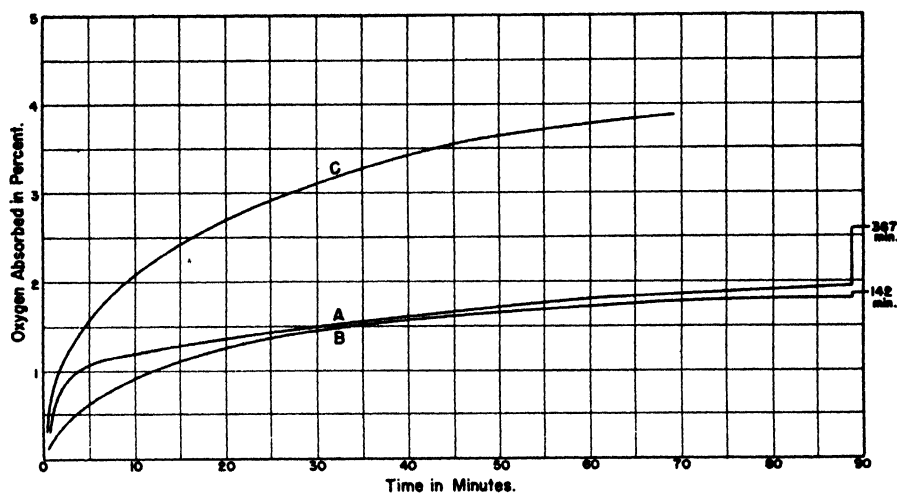


FIG. 3. Rate of oxygenation of 3-Nitro Co-Ox. A, Dry air ( $\text{Mg}(\text{ClO}_4)_2$ ),  $25^\circ$ , 700 mm. Hg.; B, oxygenation; and C, oxygenation plus hydration at 56 per cent relative humidity,  $25^\circ$ , 737 mm. Hg.

Paper XIII, Method F, was used for determining the rate of oxygenation at various temperatures,  $24^\circ$ ,  $55^\circ$ ,  $70$ – $72^\circ$ ,  $76$ – $77^\circ$ , and  $87^\circ$ . The results are shown graphically in Figure 4. The rate of oxygenation of 3-Nitro Co-Ox changed only slightly with temperature. This is particularly interesting since the rate of oxygenation of the parent, oxygen-carrying compound varies greatly with temperature.

The point at which the rate curve levels off depends markedly upon

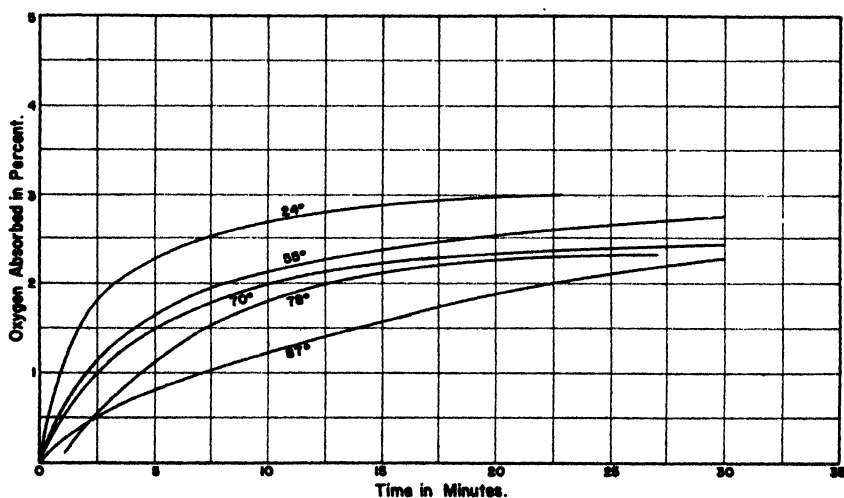


FIG. 4. Rate of oxygenation of 3-Nitro Co-Ox at various temperatures in pure dry oxygen, 736 mm. Hg.



the oxygen pressure, approaching the theoretical, oxygen-carrying capacity at the higher pressures.

Even when deoxygenated di-(2-hydroxy-3-nitrobenzal)-ethylenediimine cobalt was placed in an oxygen atmosphere at 200 pounds pressure, it did not quickly become entirely saturated with oxygen. Approximately thirty minutes was required for the last few tenths per cent of oxygen to be absorbed. However, when the compound was saturated with oxygen at 200 pounds pressure it did not lose oxygen when the oxygen pressure was released.

The role that water plays in the rate of oxygenation of di-(2-hydroxy-3-nitrobenzal)-ethylenediimine cobalt is not entirely clear. Apparently the rate of oxygenation is slightly faster in air or oxygen containing a small amount of water, that is, at relative humidities of 10 per cent or less. At the same time the compound is rendered inactive by the absorption of water. The deoxygenated compound upon absorption of water immediately turns yellow.

#### THE ABSORPTION OF MOISTURE AND OF OXYGEN BY DI-(2-HYDROXY-3-NITROBENZAL)-ETHYLENEDIIMINE COBALT

A series of weighed samples of the activated di-(2-hydroxy-3-nitrobenzal)-ethylenediimine cobalt were exposed to air at various humidities and allowed to come to equilibrium with oxygen and water. The moisture content of the air ranged from 0.2 mg. per liter, secured by using the proper concentration of sulfuric acid in a desiccator. At the end of seventy-two hours each sample was weighed, then deoxygenated and dehydrated in an electrically heated tube at 130° through which was passed a stream of dry nitrogen. The moisture evolved was collected in a U-tube filled with magnesium perchlorate. The sample was then cooled in a vacuum desiccator and weighed. The total loss in weight was the sum of the oxygen and moisture absorbed. From the weight gained by the U-tube and the total loss in weight, the amount of moisture and the amount of oxygen absorbed at each humidity was calculated. The results are summarized in Table 1.

It is apparent that the absorption of oxygen decreases markedly with

TABLE 1  
COMPETING ABSORPTION OF OXYGEN AND WATER FROM MOIST AIR BY 3-NITRO-Co-Ox

Sulfuric Acid Concentration		Water Content of Air, Mg. per liter	Oxygen Absorbed, Percentage	Water Absorbed, Percentage
Density	Percentage			
1.299	39.6	25.4	1.93	1.98
1.450	55.5	11.4	2.16	1.40
1.521	62.1	6.4	*	*
1.629	71.6	1.7	2.6	0.6
1.740	81.2	0.24	2.85	0.3

\* Sample was lost during the course of the run.

increasing water content in the air. From a comparison with the corresponding data for 3-Methoxy Co-Ox (Paper VI of this series) it is evident that the effect of moisture on the 3-nitro compound is much less. However, because of this competing absorption of water and oxygen, the air employed to oxygenate the compound when used for production of oxygen should be thoroughly dried.

In every case upon deoxygenation and dehydration at 130° the samples returned to their original weight showing that all of the moisture had been removed at this temperature. The possibility of entirely removing absorbed moisture at a much lower temperature has not as yet been definitely ascertained.

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# STUDIES ON OXYGEN-CARRYING COBALT COMPOUNDS

## VI. DI-(2-HYDROXY-3-METHOXYBENZAL)-ETHYLENEDIIMINE COBALT.

### 3-METHOXY CO-OX

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The compound di-(2-hydroxy-3-methoxybenzal)-ethylenediimine cobalt, similar in constitution to disalicylalethylenediimine cobalt (Co-Ox) but derived from 3-methoxysalicylaldehyde rather than salicylaldehyde, was found to behave as an oxygen carrier but to absorb oxygen at a rate considerably greater than that of the parent compound. This material has been designated as *3-Methoxy Co-Ox*.

The rapid rate of oxygenation of this material at atmospheric pressure in dry air gives it a signal advantage over other compounds investigated. It makes possible a considerable reduction in the time required per cycle of oxygenation and deoxygenation, and it eliminates the need for high pressure air and correspondingly heavier apparatus. These advantages are partially offset, however, by the need of refrigeration for oxygenation since the temperature at which the oxygenation rate is a maximum is near 0°. When first produced di-(2-hydroxy-3-methoxybenzal)-ethylenediimine cobalt separates as a hydrate, yellow in color and in silky micro crystals. On heating to 170° in a vacuum this hydrate, which is inactive toward oxygen, loses one molecule of water per cobalt atom and becomes maroon in color. It is then capable of absorbing one molecule of oxygen per two cobalt atoms, turning black in the process.

The deoxygenated form of the material is paramagnetic; the oxygenated form is weakly diamagnetic.

### PREPARATION

3-Methoxy Co-Ox can be prepared by a variety of methods but not all are of equal merit. For the laboratory preparation of relatively small lots, the direct mixing of the exact amounts of the three reactants, 2-hydroxy-3-methoxybenzaldehyde, ethylenediamine, and cobalt acetate, in an aqueous-alcohol medium was found best. Conditions were arranged so that the final solution was 50 to 60 per cent alcohol. Although the yield by this procedure was only about 60 per cent, the oxygen-carrying capacity of the product was consistently close to the theoretical 4.15 per cent.

Attempts were made to reduce the amount of alcohol used. With only enough alcohol to dissolve the aldehyde and with vigorous stirring and sufficient digestion at 80°, a satisfactory product was obtained. The

material was difficult to filter, however, and although the yield was 90 per cent, the oxygen-carrying capacity of the product was somewhat low, about 4.0 per cent. The product obtained was contaminated with some unreacted Schiff's base which distilled out slowly during activation.

The best method found for the large scale preparation of 3-Methoxy Co-Ox involved the preliminary preparation of the Schiff's base in about 50 per cent alcohol. Without isolating the Schiff's base, it was dissolved by the addition of a hot, sodium hydroxide solution and the cobalt derivative was precipitated by the addition of a solution of the cobalt salt buffered with acetic acid and sodium acetate, the final concentration of alcohol being about 25 per cent. Several attempts were made to eliminate all or most of the alcohol by precipitating the Schiff's base from a very dilute solution of ethylenediamine in water. It was found, however, that the large particles of the Schiff's base formed in this manner were difficult to dissolve in the hot caustic solution. When the Schiff's base was precipitated from dilute alcohol the particle size of the material was such that it dissolved immediately upon the addition of the hot sodium hydroxide solution. The minimum alcohol concentration for this step was found to be about 50 per cent. This was obtained by adding an alcohol solution of the aldehyde to a hot solution of ethylenediamine dissolved in water. The product so obtained was readily filtered and no appreciable difficulty was experienced in filtering the material after reslurrying with warm water. Occasionally, however, it was found necessary to add a small amount of alcohol to the wash solution in order to minimize peptization which rendered the final filtration difficult. The yields by this method of preparation were 85 to 90 per cent. Upon activation the material prepared in this manner carried 4.1 per cent oxygen. This method is recommended for the large scale preparation of 3-Methoxy Co-Ox and the directions are reproduced in detail below.

A number of attempts were made to prepare 3-Methoxy Co-Ox by the direct interaction of a suspension of the Schiff's base and a solution of a cobalt salt. The finely pulverized Schiff's base was digested with a solution of a cobalt salt on a steam bath with continuous, mechanical agitation for periods up to twelve hours. The compound obtained was then filtered or centrifuged, dried, and activated. The oxygen-carrying capacity of the final product approached the theoretical value of 4.15 per cent provided a sufficient period of digestion were allowed but in general the method was not highly satisfactory. In the case of 3-Nitro Co-Ox (see Paper V) this is the only method by which an active cobalt compound can be prepared.

3-Methoxy Co-Ox was also made from the sodium salt of 3-methoxysalicylaldehyde by reaction with a cobalt salt and ethylenediamine. The precipitate was difficult to filter although the final product had practically the theoretical oxygen-carrying capacity. The parent material, Co-Ox, cannot be prepared by this method.

When di-(2-hydroxy-3-methoxybenzal)-ethylenediimine cobalt was formed in the presence of pyridine it crystallized with one molecule of

pyridine per cobalt atom rather than as the hydrate discussed above. The pyridinate was stable toward drying at 100° and was inactive toward oxygen. The pyridine was expelled on heating at 170° in a vacuum, just as was the water from the hydrate, and the residual material was also active toward oxygen. As a method of preparation, however, preliminary preparation of the pyridinate was found to be unsatisfactory as the oxygen-carrying capacity of the product in no instance among a number of preparations exceeded 2.2 per cent.

#### HYGROSCOPIC CHARACTER

Both the deoxygenated and the oxygenated forms of di-(2-hydroxy-3-methoxy)-benzaethylenediimine cobalt were found to be very hygroscopic. It was hoped that the compound in the active state would absorb oxygen to its theoretical capacity even though it absorbed moisture at the same time and that the oxygen absorption might be reversible and undiminished. If true it might eliminate the necessity for thoroughly drying the air to be used in the oxygenation cycle. This hope was chimerical, however.

Both forms absorbed about 15 per cent of water on exposure to air saturated with moisture at room temperature. On subsequently heating the oxygenated form to 100° the major portion of this water was eliminated, a residual amount, about 2.3 per cent, apparently having no effect on the oxygen-carrying capacity. On subsequently warming the deoxygenated form a change in color from red to orange occurred corresponding to the formation of an inactive hydrated form; concurrently the oxygen-carrying capacity decreased markedly, disappearing completely if sufficient water was present. All of the forms were reactivated when heated to 170° in a vacuum.

The relative amounts of water and oxygen absorbed by the deoxygenated compound on exposure to air of different moisture contents was determined. The water content of the air must be reduced to at least 0.1 mg. per liter to avoid serious absorption of water by the compound during use.

#### RATE OF OXYGENATION

The rate of oxygenation of 3-Methoxy Co-Ox at various temperatures in a stream of dry air at various temperatures was determined by the gravimetric method described in the experimental work given below. The material oxygenated very slowly in air at 25°, about fifteen hours being required for saturation; it is doubtful if the theoretical capacity was ever reached under atmospheric pressure at this temperature. In pure oxygen at this temperature the compound became completely saturated. The rate of oxygenation using dry air increased remarkably as the temperature was lowered below 25°. At 0° complete saturation was reached in about ten minutes. This compares favorably with the rate obtained when pure oxygen was used at 25°. At a temperature of -70°, the rate

was very much slower than at  $0^{\circ}$ . Even at this low temperature the rate was still somewhat greater than at room temperature.

The temperature of optimum rate of oxygenation was in the range between  $12^{\circ}$  and  $-10^{\circ}$ .

The rate of oxygenation of di-(2-hydroxy-3-methoxybenzal)-ethylenediimine was considerably greater than that of the parent compound, Co-Ox, (Paper III), but somewhat slower than that of 3-Nitro Co-Ox (Paper V). It was deoxygenated at a lower temperature than either of the other compounds, however; at  $55^{\circ}$  it was completely deoxygenated at atmospheric pressure.

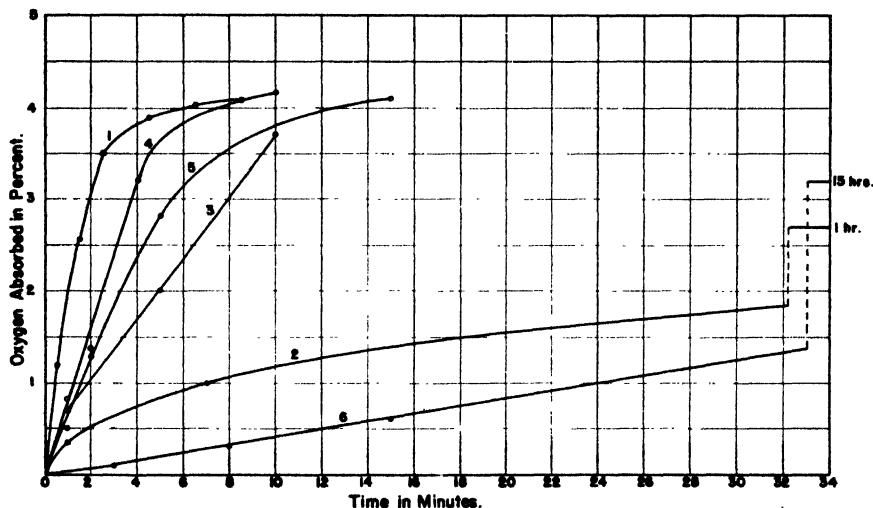


FIG. 1. Rate of oxygenation of 3-Methoxy Co-Ox at various temperatures. Curve 1: oxygen at  $25^{\circ}$ ; 2: air at  $-70^{\circ}$ ; 3: air at  $-10^{\circ}$ ; 4: air at  $0^{\circ}$ ; 5: air at  $12^{\circ}$ ; 6: air at  $25^{\circ}$ .

#### RATE OF DETERIORATION

The rate at which di-(2-hydroxy-3-methoxybenzal)-ethylenediimine cobalt deteriorated was determined using a stationary bed of material heated and cooled between  $100^{\circ}$  and  $15^{\circ}$ . The rate of deterioration was more rapid at the beginning than toward the end of the study. After 2,700 cycles of oxygenation and deoxygenation, the oxygen-carrying capacity of the material had fallen from 4.15 to 0.78 per cent. After reactivation, however, the oxygen-carrying capacity was 2.2 per cent, showing that part of the apparent deterioration was due to the absorption of water. The true deterioration then was about 50 per cent of the oxygen-carrying capacity of the compound in 2,700 cycles.

#### EXPERIMENTAL WORK

##### DI-(2-HYDROXY-3-METHOXYBENZAL)-ETHYLENEDIIMINE

The condensation of ethylenediamine and 2-hydroxy-3-methoxybenzaldehyde was best carried out in an alcohol medium from which

the Schiff's base precipitated as a bright yellow, crystalline material. The condensation was also effected in water solution but the product tended to form large lumps which dissolved very slowly when later treated with sodium hydroxide (the first operation in the formation of the cobalt derivative). The Schiff's base was recrystallized from hot, absolute alcohol and from ether; m.p.:  $161^{\circ}$ . Recrystallization was not necessary for the preparation of a satisfactory oxygen-carrying cobalt derivative. Indeed, in the procedure recommended for the large scale preparation of 3-Methoxy Co-Ox, the Schiff's base after being formed was not filtered off but dissolved in alkali and used directly.

The nitrogen in this base was determined by the Kjeldahl method beginning the digestion with dilute sulfuric acid (1 : 2). Found: 8.35, 8.36, 8.29, 8.33, 8.34 per cent N; calculated for  $C_{18}H_{20}O_4N_4$ : 8.53 per cent N, for  $C_{18}H_{20}O_4N_4 \cdot \frac{1}{2}H_2O$ : 8.31 per cent N.

DI- (2-HYDROXY-3-METHOXYBENZAL)-ETHYLENEDIIMINE COBALT  
(3-METHOXY CO-OX)

*Recommended Procedure for Small Scale Laboratory Preparation.* To 4.4 g. of a 68.7 per cent solution of ethylenediamine (0.05 mole) dissolved in 50 ml. of 95 per cent ethyl alcohol add 12.45 g. of cobalt acetate,  $Co(C_2H_3O_2)_2 \cdot 4H_2O$  (0.05 mole) dissolved in 200 ml. of 50 per cent ethyl alcohol warmed to  $60^{\circ}$ . To this solution add 15.2 g. (0.10 mole) of 2-hydroxy-3-methoxybenzaldehyde (Monsanto Chemical Company, m.p.:  $42^{\circ}$ ) dissolved in 100 ml. of 70 per cent ethyl alcohol. A clear brown solution results and in about a minute a silky, light brown precipitate forms. After thirty minutes filter the precipitate on a Buchner funnel, wash with 50 ml. of cold 50 per cent ethyl alcohol and dry in a vacuum at  $100^{\circ}$  for two hours. Pulverize the material and dry it further at  $100^{\circ}$  in a vacuum. Finally activate the yellow hydrate by heating it to  $170^{\circ}$  in a vacuum for two hours. The product is maroon in color and very hygroscopic. Yield: 60 per cent; oxygen-carrying capacity: 4.15 per cent.

*Recommended Procedure for the Large Scale Preparation of 3-Methoxy Co-Ox.* In a 20-gallon crock heat 16 l. of water to boiling and dissolve in it 0.95 l. of 68.5 per cent ethylenediamine. In another crock heat 8 l. of alcohol to boiling and dissolve in it 2.66 l. of 2-hydroxy-3-methoxybenzaldehyde. Add this hot solution of the aldehyde to the diamine solution and stir thoroughly. In another crock heat 4 l. of water to boiling and dissolve in it 0.82 kg. of sodium hydroxide and 0.41 kg. of sodium acetate. Add this solution to the crock containing the Schiff's base and stir thoroughly until all the crystalline material has dissolved. In another crock heat 8 l. of water and 0.4 l. of acetic acid to boiling and dissolve in the solution 2.5 kg. of cobalt chloride. Add this hot cobalt solution to the solution of the sodium salt of the condensation product and stir vigorously for fifteen minutes. Then allow the material to stand at least one hour before filtering. Return the filtered material to the original crock and mix thoroughly with 40 l. of water. Filter, and repeat the washing process. Suck the material as dry as possible on the funnel.

Place the mass in a drying oven so arranged that a stream of warm air sweeps over the compound. Dry at a final temperature of 110°. After the material is completely dry throughout the mass, grind up the chunks in a burr mill. Activate the material in a rotating drum surrounded by an electrically heated oven at 170–180°. Yield: 85 to 90 per cent; oxygen-carrying capacity: 4.1 per cent.

#### COMPOSITION OF 3-METHOXY CO-OX

A sample of di-(2-hydroxy-3-methoxybenzal)-ethylenediimine cobalt was carefully prepared by the direct mixing of the reactants in alcohol, by the procedure described above. The yellow, hydrated product was dried in a vacuum at 100°. It was then heated at 170° in a vacuum and the loss in weight determined to be 4.5 per cent. The theoretical loss in weight for the expulsion of one molecule of water from the monohydrate is 4.47 per cent, from a sesquihydrate: 4.37 per cent.

The cobalt in both the hydrate and the activated, oxygenated material was determined by decomposing the weighed sample by digestion with nitric acid and sulfuric acid, evaporation to fumes of sulfuric acid, and titration with ferricyanide in ammoniacal tartrate solution. Found for the yellow, hydrated material: 14.21, 14.12 per cent Co; theoretical for  $C_{18}H_{18}O_4N_2Co \cdot H_2O$ : 14.61 per cent Co, for  $C_{18}H_{18}O_4N_2Co \cdot 1\frac{1}{2}H_2O$ : 14.30 per cent Co. Found for the activated, deoxygenated material: 14.78, 14.82, 14.83, 14.80, 14.74, 14.80 per cent Co; theoretical for  $C_{18}H_{18}O_4N_2Co, anhy.$ : 15.30 per cent Co, for  $C_{18}H_{18}O_4N_2Co \cdot \frac{1}{2}H_2O$ : 14.95 per cent Co.

The nitrogen was determined by the Kjeldahl method starting with dilute sulfuric acid. Found on activated, deoxygenated material 6.93, 6.41, 6.84, 6.75, 6.60, 6.61, 6.69 per cent N; theoretical for  $C_{18}H_{18}O_4N_2Co, anhy.$ : 7.27 per cent Co; for  $C_{18}H_{18}O_4N_2Co \cdot \frac{1}{2}H_2O$ : 7.11 per cent Co.

#### ABSORPTION OF MOISTURE BY 3-METHOXY CO-OX

Weighed samples of both oxygenated and deoxygenated di-(2-hydroxy-3-methoxybenzal)-ethylenediimine cobalt were placed in a tube through which air saturated with water vapor at 30° was drawn. The oxygenated material gained from 15 to 20 per cent in weight depending on the time, ten to twenty-four hours, owing to the absorption of water. Upon heating at 100° in a vacuum most of this water was expelled but about 2.3 per cent remained. The residual material absorbed and released the same quantity of oxygen as before the absorption of water. This residual water was expelled completely at 170° in a vacuum without alteration in the oxygen-carrying capacity of the material.

In the case of the deoxygenated material the effect was quite different. A sample of the deoxygenated material which had been shown previously to carry the theoretical amount of oxygen, 4.15 per cent, upon exposure to air saturated with water vapor increased in weight to 15 per cent: no appreciable change in color occurred: This material, after having



absorbed 15 per cent water, was placed in an atmosphere of pure oxygen at 175 pounds pressure; it absorbed only 1.9 per cent oxygen. When then heated to 100° in a vacuum it retained about 2 per cent of water (the oxygen being expelled) and the subsequent oxygen-carrying capacity was only 2.9 per cent. This material was completely reactivated when heated in a vacuum to 170°.

The failure of the deoxygenated material to change color upon the absorption of even 15 per cent of water on exposure to moist air is interesting. In a number of instances a change was observed when the material was heated somewhat above room temperature, the color changing from the reddish brown of the deoxygenated material to the yellow color of the hydrate. It appeared that a definite chemical reaction took place somewhat above 60° to form the inactive hydrate.

In another series of experiments, samples of active, deoxygenated di-(2-hydroxy-3-methoxybenzal)-ethylenediimine cobalt were exposed to air of various moisture contents and the amounts of oxygen and of water absorbed determined. The oxygen-carrying capacity of the compound used was 4.1 per cent. Samples of about 2 g. were weighed into identical nickel boats, deoxygenated at 170° in a vacuum for fifteen minutes, cooled in an evacuated desiccator, then quickly weighed and placed in desiccators of the same size containing suitable concentrations of sulfuric acid to adjust the moisture content of the air. The concentrations of sulfuric acid were determined by specific gravity measurements using a pycnometer. The moisture content of these solutions was calculated in mg. of water per liter, from vapor pressure data for sulfuric acid solutions taken from *International Critical Tables*, Vol. III, p. 303. The samples were left in the desiccators sixty hours. The total gain in weight—that is, the sum of the oxygen and the water absorbed—was measured and the water present was determined by heating the samples in an electrically heated tube to 170° with a stream of dry nitrogen flowing through the tube to sweep the water into a weighed U-tube containing anhydrous magnesium perchlorate. The data is summarized in Table 1.

From these experiments it was evident that if 3-Methoxy Co-Ox was to be employed for the production of oxygen from air, the moisture in

TABLE 1  
COMPETING ABSORPTION OF OXYGEN AND WATER FROM MOIST AIR BY 3-METHOXY CO-OX

Sulfuric Acid Concentration		Water Content of Air, Mg. per liter	Oxygen Absorbed, Percentage	Water Absorbed, Percentage
Density	Percentage			
1.299	39.6	30.2	0.5	15
1.450	55.5	25.4	0.96	10.6
1.521	62.1	11.4	2.5	4.0
1.629	71.6	6.4	2.68	2.7
1.740	81.2	1.7	2.70	2.1
		0.24		0.3

the air used for the oxygenation must be reduced to below 0.2 milligrams of water per liter of air. This can be done readily by employing potassium hydroxide as the drying agent.

As a further proof that the hydrate is actually formed when moisture is absorbed and the material heated, about 20 g. of deoxygenated di-(2-hydroxy-3-methoxybenzal)-ethylenediimine cobalt was placed in a 600 ml. beaker and covered with 250 ml. of distilled water. The mixture was stirred and heated gradually to 90°, and then allowed to digest on a steam plate overnight. The color of the material changed from reddish brown to the familiar yellow color of the hydrate. The material was filtered on a Buchner funnel and dried in a vacuum at 100° for twelve hours. A portion of this material was pulverized and a weighed sample activated at 170° under a vacuum. The loss in weight upon activation was 4.65 per cent: theoretical for one molecule of water per cobalt atom: 4.68 per cent.

#### RATE OF OXYGENATION OF 3-METHOXY CO-OX

The method employed in the determination of the rate of oxygenation consisted in placing a sample of material in a U-tube, packed well with glass wool to prevent loss of compound, and then oxygenating the sample by passing a rapid stream of air through the tube and observing the gain in weight. The compound was deoxygenated by placing the U-tube in a beaker of hot water and passing a slow stream of nitrogen through the tube. Rates of oxygenation at various temperatures were obtained by placing the U-tube in a liquid bath at the desired temperature and passing dry air through the tube. The temperature of -70° was obtained by using a bath of chloroform and pieces of solid carbon dioxide.

The results of the various rate measurements are shown graphically in Figure 1. Measurements of the rate of oxygenation of 3-Methoxy Co-Ox in pure oxygen are reported in Paper XII, Figure 2.

#### RATE OF DETERIORATION OF 3-METHOXY CO-OX

About 40 g. of powdered 3-Methoxy Co-Ox was placed in a brass tube 1.2 cm. in diameter and 70 cm. long, fitted with a jacket through which cold water or steam could be passed. With cooling water at 10° to 15° in the jacket the material was oxygenated by the passage through the tube of a slow stream of air, about 3 liters per minute, the pressure of the entering air and the exit air being 80 and 20 pounds per square inch gauge, respectively. The air used was thoroughly dried by passage through a mechanical water trap, a drying tower of potassium hydroxide and a tower of anhydrous magnesium perchlorate. About six minutes was required for oxygenation after which the cooling water was drained from the jacket and steam at atmospheric pressure admitted to effect the deoxygenation. The deoxygenation was carried out with the air stream stopped and the tube open to the atmosphere. The entire mechanism was operated mechanically by a suitable timing device.

The capacity of the material was determined at intervals by removing a portion of the compound from the tube. This sample was deoxygenated by heating to 100° in a vacuum and its oxygen-carrying capacity determined. The rate of deterioration is shown in Table 2.

TABLE 2  
RATE OF DETERIORATION OF 3-METHOXY CO-OX

Number of Cycles	Capacity, Percentage	Rate of Deterioration, Percentage of Original Capacity per 100 Cycles
0.....	4.15	.....
336.....	3.71	3.18
1,645.....	2.18	2.82
2,700.....	1.84	0.78

The deterioration proceeded much more rapidly at the beginning of the test. Like the parent oxygen-carrying compound, 3-Methoxy Co-Ox is an excellent heat insulator. It may have been that with a tube of the size used in this test that only the outside layer of the material was fully heated and cooled during the cycle. If this were the case it might be reasonably expected that the outside layer would deteriorate more rapidly and after rather complete deterioration would protect the inner layers of the material from heat changes to such an extent that the rate of deterioration would become much slower.

After the final capacity test the entire remaining material was heated to 170° in a vacuum. Its oxygen-carrying capacity was then 2.2 per cent. It appeared, therefore, that hydration had taken place in some way. Since the air was dried over anhydrous magnesium perchlorate, the more reasonable source of this moisture is the combustion of the organic portion of the molecule.

## STUDIES ON OXYGEN-CARRYING COBALT COMPOUNDS

### VII. DI-(2-HYDROXY-3-ETHOXYBENZAL)-ETHYLENEDIIMINE COBALT AND HIGHER 3-ALKOXY ANALOGUES. 3-ETHOXY CO-OX AND CO-OX SS

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In Paper VI of this series it was reported that di-(2-hydroxy-3-methoxybenzal)-ethylenediimine cobalt, 3-Methoxy Co-Ox, absorbed oxygen much more rapidly than the parent oxygen-carrying compound, Co-Ox. Naturally it became of interest to learn the effect of lengthening the alkoxy chain.

2-Hydroxy-3-ethoxybenzaldehyde, 2-hydroxy-3-*n*-propoxybenzaldehyde and 2-hydroxy-3-*n*-butoxybenzaldehyde were synthesized from the corresponding *o*-alkoxyphenols by the Duff reaction (1). *o*-Ethoxyphenol was prepared by the action of diethylsulfate on pyrocatechol in alkaline solution. The higher *o*-alkoxyphenols were prepared by the action of the alkyl bromide on pyrocatechol. Various factors affecting the yields from the Duff reaction were investigated in connection with this work and were reported by Liggett and Diehl (2).

Like the Schiff's bases of salicylaldehyde and of 2-hydroxy-3-methoxybenzaldehyde with ethylenediamine, the Schiff's base with the higher 3-alkoxy aldehydes are also crystalline compounds, yellow in color, with melting points decreasing with increasing length of the alkoxy chain.

Di-(2-hydroxy-3-ethoxybenzal)-ethylenediimine cobalt, designated 3-Ethoxy Co-Ox, proved to be active toward oxygen and to absorb 3.80 per cent oxygen, the theoretical amount for one molecule of oxygen per two cobalt atoms. When first formed it precipitated as a yellow or maroon-colored material depending on the prevailing conditions. The yellow material was apparently a hydrate but lost its water so easily that it could not be brought to constant weight for analysis. It yielded the maroon-colored material on drying. The maroon material absorbed oxygen directly, turning black during the process.

Numerous preparations of the 3-ethoxy compound were made with object of devising a satisfactory method for its preparation. The amount of alcohol used as solvent, the total volume of solution, the amount of excess acid added, and the time of digestion of the precipitate were varied systematically. Conditions were found for the preparation of easily filterable material of high capacity in good yield.

As was pointed out in Paper VI, 3-Methoxy Co-Ox is extremely hygroscopic and the absorbed water is expelled only when the compound

is heated to  $170^{\circ}$ . 3-Ethoxy Co-Ox was found to be much less hygroscopic. It did, however, absorb water along with oxygen from moist air and the oxygen-carrying capacity was decreased by the water absorbed. The absorbed water was expelled when the material was heated in a stream of nitrogen at  $100^{\circ}$ , the oxygen-carrying capacity returning as the water was eliminated. It was thought possible to operate the oxygenation-deoxygenation cycle with moist air since the water was found to be eliminated from the compound at a temperature as low as  $100^{\circ}$ . Thus, although water and oxygen were simultaneously absorbed during oxygenation with moist air, the water might be expelled during the deoxygenation step at  $100^{\circ}$ . When tried it was found, however, that although the water was expelled from the compound at  $100^{\circ}$ , the amount of oxygen produced was not sufficient to sweep all of the water from the material. The amount of water in the material gradually increased and although the water reached a rather steady value the oxygen-carrying capacity was somewhat too low for practicable use. This was the case using air with relative humidities of 25 per cent and 75 per cent. Even using air dried over solid potassium hydroxide (0.002 milligram of water per liter) there was a steady accumulation of about 0.01 per cent water per cycle. It appeared therefore that if a deoxygenation temperature of  $100^{\circ}$  was used that it was necessary to either dry the air carefully or to periodically sweep the water from the compound with air or nitrogen at  $100^{\circ}$ . Air having a water content of 5 milligrams per liter could be used if the operation were occasionally stopped and the water expelled by heating in a stream of air or nitrogen or the cycle so arranged that a hot air flushing operation followed the deoxygenation.

This experiment was repeated using a deoxygenation temperature of  $130^{\circ}$ . Under these conditions the water expelled from the compound was all converted to steam and removed from the vicinity of the material. Air of 25 per cent humidity was used; the residual water after deoxygenation (ten minutes,  $130^{\circ}$ ) assumed a steady value of 0.4 per cent and the oxygen-carrying capacity was essentially unaffected, remaining at about 3.6 per cent. This appeared to be a very practical way to operate the cycle on moist air.

This experiment was again repeated carrying out the deoxygenation at  $100^{\circ}$  under reduced pressure (220 mm.). The gradual diminution of the oxygen-carrying capacity of the material indicated that reducing the pressure alone was not sufficient. Although no experiment was made, undoubtedly deoxygenation at  $130^{\circ}$  under reduced pressure would be successful.

An interesting possibility is that of making the absorption of oxygen an exchange reaction with water. The heat of reaction would then be greatly decreased, being the difference between the heat of oxygenation and the heat of dehydration, and the rate of deterioration of the compound might be consequently decreased. Experiments indicated that such an exchange reaction did actually take place but was far from complete and apparently not very practical.

Di-(2-hydroxy-3-*n*-propoxybenzal)-ethylenediimine cobalt was also prepared and found to function as an oxygen carrier. In an atmosphere of oxygen in the differential manometric testing apparatus, the rate of oxygenation of this material was found to be the greatest of any of the compounds studied. Attempts to determine the rate of oxygenation in air at atmospheric pressure were thwarted owing to a tendency of the material to pack and to prevent the passage of air. The compound was exceptionally hygroscopic.

The compound di-(2-hydroxy-3-*n*-butoxybenzal)-ethylenediimine cobalt was synthesized and found to absorb oxygen at an exceptionally fast rate. Because of its extraordinary speed of oxygenation this material was given the name Co-Ox SS (Co-Ox Super Speed). The best preparations of this cobalt compound were made by dissolving the Schiff's base in alcohol and adding an aqueous solution of cobalt acetate so that the final alcohol concentration was about 40 per cent. The compound prepared this way carried 3.3 per cent oxygen, the theoretical value being 3.42. Other methods of preparation involving aqueous solutions of the sodium salt of the Schiff's base or suspensions of the Schiff's base in water yielded material of considerably lower capacity. The material formed from the dilute alcohol solution was a yellow hydrate, similar in appearance to the corresponding methoxy compound. Unlike the methoxy compound, however, the butoxy compound became active, losing water, at temperatures from 100–125°. The rate of oxygenation was determined at 0°, 15°, 30°, 45°, and 50°. The rate of oxygenation was found to be practically independent of the temperature over this range. At these temperatures oxygenation was essentially complete in 60 seconds. The deoxygenation temperature was found to be about 80°.

## EXPERIMENTAL WORK

### A. DI-(2-HYDROXY-3-ETHOXYBENZAL)-ETHYLENEDIIMINE COBALT. 3-ETHOXY CO-OX

In the earliest reference to the mono-alkyl ethers of pyrocatechol, Merck (3) discussed the preparation of *o*-methoxyphenol (guaiacol, guajakol) from *o*-dimethoxybenzene (veratrole) and suggested that the method would be applicable to the preparation of other monoalkoxyphenols. The method involved treatment of the dimethoxy compound with alkali, dilution, and steam distillation. The dimethoxy compound did not distill and the yield was almost quantitative. The Merck procedure was mentioned somewhat later (4) and the boiling points of several monoethers were reported. However, no details are given nor does any subsequent literature give experimental directions.

During 1932 and 1933 *o*-ethoxyphenol was prepared by several methods and its physical properties reported: the diazotization and subsequent hydrolysis of *o*-phenetidine (5); from the diethyl ether by a Grignard reaction (6); and by the action of the alkyl halide on pyrocatechol in the presence of potassium hydroxide or carbonate (7).

It was thought that the conversion of pyrocatechol to the monoether by diethylsulfate offered a more convenient source of the material than the methods just mentioned. This proved to be the case and during the course of a number of preparations various factors influencing the reaction were varied to determine the optimum conditions.

#### O-ETHOXYPHENOL

*Recommended Procedure.* In a 1 l. three-necked flask provided with a reflux condenser, a motor driven stirrer, a dropping funnel, and a gas inlet tube, mix 1 mole (110 g.) of pyrocatechol with 200 ml. of water. In a separate flask dissolve 1 mole (40 g.) of sodium hydroxide in 100 ml. of water. Heat the pyrocatechol solution to boiling with constant stirring and pass a stream of nitrogen through the flask. When the solution is refluxing evenly and all of the air has been displaced from the flask, add the sodium hydroxide through the dropping funnel. Rinse the dropping funnel with a little distilled water and then pour 158 g. (4 g. excess) of diethylsulfate into it. Add this diethylsulfate slowly to the refluxing solution over a period of forty-five minutes. Reflux the final mixture for one hour, cool and separate the top (oily) layer. Vacuum distill this oil collecting a 5° fraction; at 10 mm. pressure the oil will come over between 100° and 105°. If the product is slightly impure it will turn yellow after a time. A redistillation will purify it. Yield: 50 per cent.

In case a great deal of oxidation of the alkaline pyrogallol has taken place the solution may be too dark to enable a separation of the oily layer. In this case the entire solution may be acidified and then steam distilled. The oil may be then separated from the distillate and then vacuum distilled.

Reported for *o*-ethoxyphenol: b.p.: 211.1°/720.85 mm. (8), 74–76°/4 mm. (7),  $D^{25}_D$ : 1.0903 (6);  $N^{25}_D$  1.5224 (8).

#### 2-HYDROXY-3-ETHOXYBENZALDEHYDE

This aldehyde was prepared from *o*-ethoxyphenol by the Duff reaction (2). Yield: 10.6 per cent; m.p.: 60–62°, reported (9): 64–65°. Later a technical grade of the aldehyde was obtained from the Monsanto Chemical Company; m.p. after vacuum distillation: 63–64°.

#### DI-(2-HYDROXY-3-ETHOXYBENZAL)-ETHYLENEDIIMINE

The condensation of the aldehyde and ethylenediamine was effected in a hot alcohol solution using a slight excess of the diamine. The yellow Schiff's base was recrystallized from dilute alcohol; m.p.: 132°.

#### DI-(2-HYDROXY-3-ETHOXYBENZAL)-ETHYLENEDIIMINE COBALT

To 4 g. of di-(2-hydroxy-3-ethoxybenzal)-ethylenediimine dissolved in 210 ml. of alcohol was added 2.81 g. of cobalt acetate dissolved in 45 ml.

of hot water. A gray-gold precipitate appeared immediately. It was filtered off and dried in a vacuum desiccator; the color changed to a dark brown. When heated in a vacuum to  $100^{\circ}$  it became brick red in color and active toward oxygen, absorbing 3.3 per cent oxygen. The theoretical capacity for the compound, absorbing one molecule of oxygen per two cobalt atoms, is 3.80 per cent. The oxygen-carrying capacity of this material increased to 3.61 per cent when it was heated in a vacuum at  $160^{\circ}$ . At  $180^{\circ}$  slight decomposition occurred and the capacity dropped.

By using the sodium salt of the Schiff's base and carrying out the reaction in a hot solution the deep red, active form rather than the hydrate was obtained and the oxygen-carrying capacity of the material after drying in a vacuum at  $100^{\circ}$  was exactly the theoretical 3.80 per cent. Numerous preparations showed that the best product was that in which the red colored material was obtained directly. The best preparations were obtained from a solution of about 40 per cent alcohol and with 15 to 20 milliliters of liquid per gram of aldehyde. When less than 40 per cent alcohol was employed the precipitate was buff colored, flocculent, and difficult to filter. The difficulty in filtering increased on washing the precipitate with water. The preparations in less than 40 per cent alcohol also had slightly lower capacities. The capacity in most cases was above 3.50 per cent, however, and this would probably not be a serious disadvantage in the commercial preparation of the material. Filtration was easier when a much larger volume of solution was used (70 ml. per g. of aldehyde) but the buff colored hydrated material was obtained and the oxygen-carrying capacity of the product was always somewhat less than the theoretical value. The best preparations were obtained when an excess of cobalt salt was used and when some acetic acid was added.

#### RECOMMENDED PROCEDURE FOR THE PREPARATION OF 3-ETHOXY CO-OX

In a 4 l. beaker, dissolve 170 g. of 2-hydroxy-3-ethoxybenzaldehyde (light yellow solid, m.p.:  $60-64^{\circ}$ ) in 600 ml. of water and 1,000 ml. of alcohol. Heat this solution to boiling. In a separate beaker, dissolve 53 g. of 68.3 per cent ethylenediamine (or the equivalent) in 200 ml. of water and 200 ml. of alcohol. Add this rapidly to the hot solution of the aldehyde, stirring vigorously. The yellow, crystalline Schiff's base precipitates. Dissolve 40 g. of sodium hydroxide and 40 g. of sodium acetate in 500 ml. of hot water. Add this to the mixture of the mother liquor and the Schiff's base and heat until all the crystals have dissolved. In a separate beaker, heat 400 ml. of water and 68 ml. of glacial acetic acid. Dissolve 143 g. of cobalt chloride in this solution and then add it to the hot solution of the Schiff's base with vigorous stirring. Keep the solution at the boiling point for ten minutes, stirring constantly. Then allow the precipitate to cool and settle. Filter on a Buchner funnel, draw as much liquid as possible out of the precipitate by suction and then wash twice with water to remove excess acetic acid, sodium acetate, and cobalt chloride. After drawing out as much water as possible, dry in an oven at  $110^{\circ}$ .



## RATE OF OXYGENATION OF 3-ETHOXY CO-OX

The rates of oxygenation of di-(2-hydroxy-3-ethoxybenzal)-ethylene-diimine cobalt in air were determined using the *Gravimetric Rate Apparatus* described in Paper XIV (*Method D*). The results are shown in Figure 1. The rate of oxygenation in pure oxygen was also determined; these results are reported in Paper XII, Figure 3.

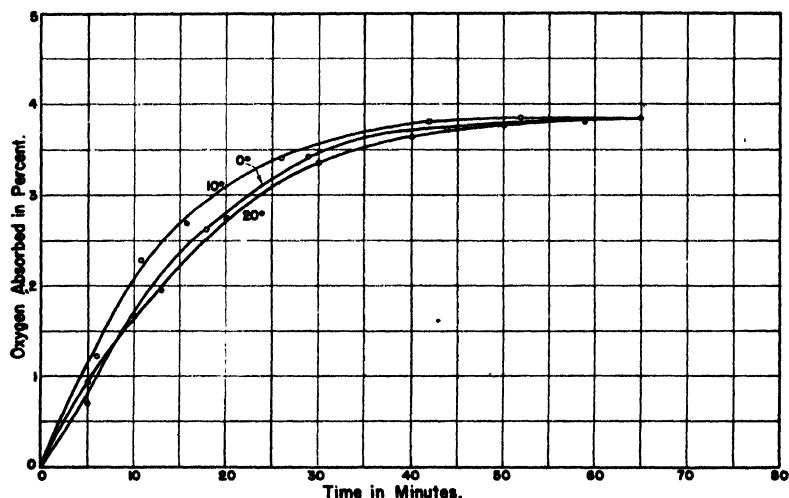


FIG. 1. Rate of oxygenation of 3-Ethoxy Co-Ox in air at various temperatures.

HYGROSCOPICITY OF DI-(2-HYDROXY-3-ETHOXYBENZAL)-  
ETHYLENEDIIMINE COBALT

Samples of deoxygenated di-(2-hydroxy-3-ethoxybenzal)-ethylene-diimine cobalt were placed in each of four desiccators each containing sulfuric acid of different concentration and allowed to stand for about a week. The gain in weight was determined in each case and the samples were analyzed for water by heating the sample at 100° in a stream of nitrogen and collecting the water in a drying tube containing anhydrous magnesium perchlorate. The amount of oxygen absorbed was thus obtained by difference. The results are shown in Table 1.

A comparison of these data with those for 3-Methoxy Co-Ox, Paper

TABLE 1  
SIMULTANEOUS ABSORPTION OF OXYGEN AND WATER FROM MOIST AIR BY 3-ETHOXY CO-OX

Sulfuric Acid Concentration, Percentage	Water Content of Air, Mg. per liter	Oxygen Absorbed, Percentage	Water Absorbed, Percentage
40.....	19.44	3.1	2.3
60.....	5.8	1.8	2.2
71.6.....	1.4	0.6	2.6
81.2.....	0.19	0.3	3.2

VI, shows that 3-Ethoxy Co-Ox is considerably less hygroscopic; thus, with air containing 2.0 mg. of water per liter, the methoxy compound absorbed 2.05 per cent water while the ethoxy compound absorbed only 0.8 per cent.

#### CYCLIC OPERATION USING MOIST AIR

Although, as shown above, water is absorbed simultaneously with oxygen from moist air by di-(2-hydroxy-3-ethoxybenzal)-ethylenediimine cobalt, it appeared possible to still operate the oxygenation-deoxygenation cycle on moist air by carrying out the deoxygenation at a temperature of 100°, since the water was expelled at this temperature. The apparatus used to test this is shown in Figure 2.

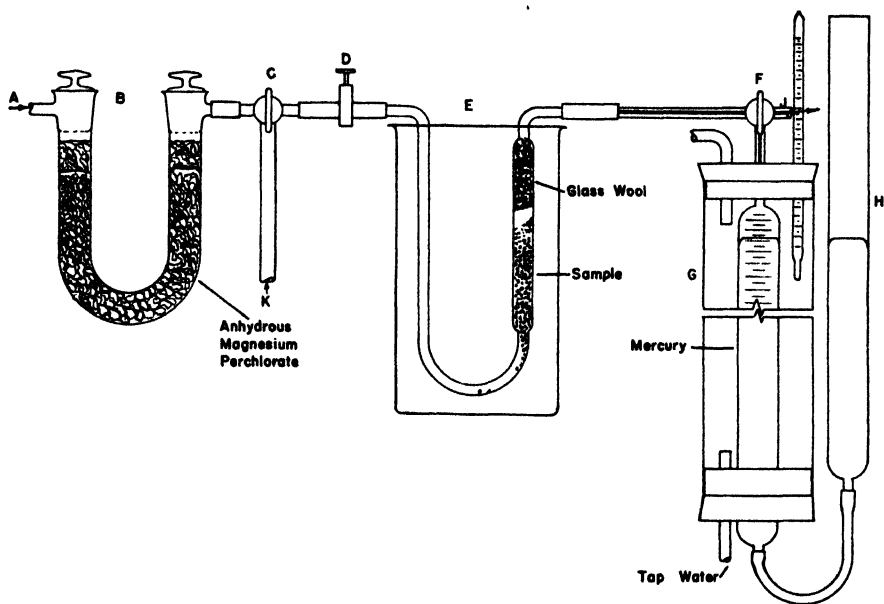


FIG. 2. Apparatus for investigation of the cyclic operation of 3-Ethoxy Co-Ox using moist air.

Nitrogen gas was admitted at inlet A and passed through the drying tube B containing anhydrous magnesium perchlorate. Inlet K was connected to a source of moist air. The humidity of the air was adjusted by bubbling it through a solution of sulfuric acid in a carboy. The moisture content of the air leaving this acid was determined by passing a measured volume of the air through a weighed magnesium perchlorate drying tube. The sample was placed in U-tube E which was immersed in a beaker of water. Outlet J was connected to a water aspirator. The gas buret G was enclosed in a water jacket through which tap water was circulated. Mercury was used as the retaining liquid in the gas buret.

The sample in U-tube E was dried to constant weight by heating the water in the bath to 100° with a stream of nitrogen passing over the

material. A blank was then determined in the following manner: (1) U-tube *E* was cooled to exactly 20° by bringing a large beaker of water around it. About twenty minutes was allowed for the temperature to reach equilibrium. The tube was flushed continuously with dry nitrogen during this period. (2) Pinch clamp *D* was closed and the mercury level in gas buret *G* was adjusted to the zero reading. Then the system was connected to gas buret *G* through stopcock *F*. (3) The U-tube was immersed in boiling water and kept at 100° for twenty minutes. (4) The quantity of gas collected in gas buret *G* was measured and the temperature of the gas and the barometric pressure were recorded. (5) The volume was corrected to standard conditions of temperature and pressure. This value is a blank buret reading due to the expansion of the gases of the system for the temperature change of from 20 to 100°.

With the sample in U-tube *E* completely deoxygenated and dried to constant weight, the apparatus was ready for the experiment which was carried out as follows: (1) Moist air was drawn through the system by opening *C* and *D* and attaching a water aspirator at *J*. The U-tube was kept in a beaker of water at 20°. (2) After drawing the air through the apparatus for ninety minutes the suction was turned off and the U-tube was dried and weighed. Then pinch clamp *D* was closed tightly, the mercury level was adjusted to zero, and stopcock *F* was opened to the buret. (3) The U-tube was immersed in a beaker of boiling water and the oxygen was collected in the buret. The water bath was kept boiling for twenty minutes to be certain of complete deoxygenation. The buret reading and temperature were recorded. (4) The stopcock *F* was closed, pinch clamp *D* was opened, and stopcock *C* was adjusted to open the system to the nitrogen train. Then, the U-tube was cooled to room temperature and nitrogen gas allowed to leak into the U-tube. When the U-tube was cool, it was weighed. (5) The stopcock *C* was opened, the aspirator turned on and the oxygenation step was begun again.

Following this procedure, which was repeated exactly each cycle as outlined above, this experiment was performed using air having a water content of 13 milligrams per liter. It was found necessary to allow ninety minutes for complete oxygenation. A temperature of 100° was used for deoxygenation and twenty minutes was required. The cycle was repeated ten times and from the data collected the quantities of moisture and oxygen absorbed were calculated. The results are given in Table 2. It will be seen that the amount of water absorbed increased to a maximum of 5.7 per cent and that the amount of oxygen carried decreased progressively. Evidently the oxygen produced was insufficient to sweep out all the water and the cycle cannot be operated efficiently under these conditions. The accumulated water was easily and completely removed, following the completion of the experiment, by sweeping nitrogen over the material at 100°.

Following the same procedure outlined above, the experiment was repeated using air having a water content of 6 milligrams per liter. The decrease in the oxygen-carrying capacity was much less and the amount of water absorbed was also much less. In this experiment the water was

TABLE 2  
CYCLIC OXYGENATION AND HYDRATION OF 3-ETHOXY CO-OX WITH MOIST AIR  
Moisture content of air: 13 mg. per liter.  
90 minute oxygenation and 20 minute deoxygenation.

Cycle	Oxygen Absorbed, Percentage	Water Absorbed During Oxygenation (accumulated), Percentage	Water Remaining After Deoxygenation (accumulated), Percentage	Water Expelled During Deoxygenation Percentage
1.....	2.85	2.6		
2.....	2.64	3.2	1.8	1.4
3.....	2.25	4.3	3.2	0.8
4.....	1.47	4.8		
5.....	1.15		3.8	
6.....	1.33	5.3	4.5	0.8
7.....	1.07	5.2	3.9	1.2
8.....	1.08	5.6	4.7	0.9
9.....	0.95	5.7	5.1	0.7
10.....	0.90	5.7	4.4	1.3

flushed away after each five cycles but the data indicate the behavior toward air of this humidity (see Table 3).

TABLE 3  
CYCLIC OXYGENATION AND HYDRATION OF 3-ETHOXY CO-OX WITH MOIST AIR  
Moisture content: 6 mg. per liter.  
30 minute oxygenation and 30 minute deoxygenation.

Cycle	Oxygen Absorbed, Percentage	Water Remaining After Deoxygenation, Percentage
1.....	3.22	
2.....	3.70†	0.82
3.....	3.22	.80
4.....	3.12	.86
5.....	3.05	
F*.....		
6.....	3.14	
7.....	3.28	
8.....	2.98	
9.....	3.18	
10.....	3.18	.98
F*.....		
11.....	3.22	
12.....	2.96	
13.....	3.08	
14.....	3.22	1.0
F*.....		
15.....	2.8	
16.....	2.98	
17.....	3.05	
18.....	3.00	
19.....	3.22	
20.....	3.70†	1.0
F*.....		

\* Flushed with nitrogen and all water removed.

† 3 hours' oxygenation.

The experiment was also repeated using air dried over walnut size potassium hydroxide which leaves 0.002 milligram of water per liter. The compound absorbed 0.01 per cent water on each cycle. At the end of thirteen cycles the accumulated water was readily expelled by passing dry nitrogen over the material at 100°. The oxygen-carrying capacity was decreased by about 0.3 per cent during the thirteen cycles. The capacity as determined by weight and by the volume of expelled oxygen checked on each cycle. This indicated that no measurable amount of water was driven off during deoxygenation. A few milligrams of sample slowly escaped through the glass wool plug in the U-tube and was lost during the experiment. It was not enough to affect the amount of oxygen absorbed, but it was enough to affect the weight and consequently the calculation of water present before and after deoxygenation. It is believed therefore that these percentages are somewhat low for the last few cycles. It is evident that the use of moist air could not be made practical simply by carrying out the deoxygenation step at a reduced pressure.

Following the same procedure, the experiment was repeated using air having a water content of 6.4 milligrams per liter but carrying out the deoxygenation at 130°. The data are given in Table 4. At the higher temperature the water was much more completely removed. Operation with moist air using 130° deoxygenation temperature appears practicable.

The experiment with moist air was performed again following the same procedure except that deoxygenation was carried out at 100° and at reduced pressure, 220 mm. The air used had a moisture content of 6.4

TABLE 4  
CYCLIC OXYGENATION AND HYDRATION OF 3-ETHOXY CO-OX WITH MOIST AIR  
AND WITH DEOXYGENATION AT 130°

Moisture content of air: 6.4 mg. per liter.  
90 minute oxygenation and 10 minute deoxygenation.

Cycle	Oxygen Absorbed, Percentage	Water Present After Oxygenation, Percentage	Water Remaining After Deoxygenation * (accumulated), Percentage
1.....	3.59	.....	0.20
2.....	3.59	0.24	0.29
3.....	3.59	0.23	0.45
4.....	3.62	0.30	0.47
5.....	3.62	0.28	0.53
6.....	3.62	0.39	0.43
7.....	3.61	0.37	0.55
8.....	3.61	0.37	0.54
9.....	3.56	0.39	0.54
10.....	3.59	0.36	0.53

\* The fact that the total water remaining after deoxygenation (Column 4) is always greater than total water before deoxygenation (Column 3) was probably due to absorption of oxygen during the cooling of the sample for weighing. If this is true, the values in Column 4 give both water plus oxygen remaining in the U-tube, and therefore these values have no significance.

milligrams per liter and oxygenation was carried out at 27° over a ninety minute period. In order to obtain the reduced pressure for deoxygenation a 150 cm. mercury-filled collecting tube was used. The low pressure was obtained by keeping the leveling bulb about 520 millimeters lower than the height of the mercury in the collecting tube. The gas was collected under a pressure of 220 millimeters and then transferred to a gas buret where it was measured at atmospheric pressure. The data is tabulated on Table 5. It can be seen that the oxygen-carrying capacity decreased quite markedly in 13 cycles.

TABLE 5

CYCLIC OXYGENATION AND HYDRATION OF 3-ETHOXY CO-OX WITH MOIST AIR AND WITH DEOXYGENATION AT 100° AND UNDER 220 MM. PRESSURE

Moisture content of air: 6.4 mg. per liter.  
90 minute oxygenation and 10 minute deoxygenation.

Cycle	Oxygen Absorbed, Percentage	Water Present After Oxygenation (accumulated), Percentage	Water Remaining After Deoxygenation (accumulated), Percentage
1.....	3.69	0.38	0.26
2.....	3.50	0.60	0.27
3.....	3.46	0.56	0.22
4.....	3.47	0.51	0.20
5.....	3.49	0.46	0.47
6.....	3.48	0.78	0.27
7.....	3.50	0.63	0.27
8.....	3.44	0.75	0.33
9.....	3.44	0.78	0.36
10.....	3.36	0.70	0.26
11.....	3.37	0.59	0.15
12.....	3.38	0.55	0.11
13.....	3.29	0.59	0.18

#### DISPLACEMENT OF WATER OF HYDRATION OF 3-ETHOXY CO-OX BY OXYGEN

It was of some interest to determine whether oxygen would displace the water from the hydrated form of di-(2-hydroxy-3-ethoxybenzal)-ethylenediimine cobalt. A definite quantity of water was introduced into the sample of di-(2-hydroxy-3-ethoxybenzal)-ethylenediimine cobalt by passing moist nitrogen gas over it. The water content of the nitrogen was adjusted by bubbling it through 30 per cent sulfuric acid solution. The nitrogen was passed through the sample in a U-tube (Fig. 1) until the compound had absorbed the desired amounts of water as determined by the gain in weight. Dry oxygen was then passed over the compound until constant weight was obtained (about one hour) indicating complete oxygenation. After this the temperature was raised to 100° and the oxygen was collected in the gas buret. By weighing the sample before and after oxygenation, the quantity of moisture introduced and the quantity expelled could be measured with reasonable accuracy. The experiment

TABLE 6  
EFFECT OF DRY OXYGEN ON A PARTIALLY HYDRATED SAMPLE OF 3-ETHOXY CO-OX

Temperature of Oxygenation	Initial Water Content, Percentage	Oxygen Absorbed (determined by measuring volume of oxygen evolved), Percentage	Water Expelled During Oxygenation, Percentage	Water Expelled During Deoxygenation, Percentage	Water Remaining After Deoxygenation, Percentage
10°.....	4.67	0.75	0.11	1.81	2.70
20°.....	4.31	1.79	1.38	*	2.93
40°.....	4.90	0.71	0.85	0.73	3.32

\* Experimental error.

was performed at 10°, 20°, and 40°. The results are tabulated in Table 6. Apparently the exchange reaction does take place to a certain extent.

B. DI-(2-HYDROXY-3-*N*-PROPOXYBENZAL)-ETHYLENEDIIMINE COBALT  
*o-n*-PROPOXYPHENOL

A quantity of 110 g. of pyrocatechol was placed in a 3-necked flask equipped with a stirrer, a reflux condenser, a dropping funnel, and a gas inlet tube. The air was flushed out of the flask with carbon dioxide gas and then the pyrocatechol was dissolved in a solution of 40 g. of sodium hydroxide in 300 ml. of water at about 50°. The solution was heated to refluxing and 170 g. of propyl iodide was added slowly over a period of one hour. Refluxing was continued for three hours. By this time the solution had become very dark and the layers in the solution were not easily distinguished even after acidifying the solution and allowing it to stand for some time. Therefore the entire solution was steam distilled. About 80 g. of oil was obtained which was dried over anhydrous calcium sulfate and vacuum distilled. Yield: 45 g.; b.p.: 112–115°/15 mm. The water layer of the steam distillate was extracted thrice with ether and the ether distilled off yielding 4 g. further of the propoxyphenol. The total yield was thus about 33 per cent. B.p.: 225–227°/740 mm., reported (4): 223–226°/760 mm.;  $n_D^{25}$ : 1.5176, reported (6): 1.5176;  $D_4^{25}$ : 1.0461, reported (6): 1.0523. The compound was soluble in dilute sodium hydroxide, the solution turning green rapidly in air.

2-HYDROXY-3-*n*-PROPOXYBENZALDEHYDE

A mixture of 35 g. of boric acid and 150 g. of glycerol was heated to 170° for twenty minutes. Then 25 g. of hexamethylenetetramine and 25 g. of *o-n*-propoxyphenol were added. The mass was stirred well and maintained at 150–155° for fifteen minutes. The reaction was quite vigorous and cooling was necessary at times to keep the temperature in this range. Finally the mass was cooled, and a solution of 30 ml. of sulfuric acid in 100 ml. of water added. Steam distillation yielded a large volume of cloudy

solution with some yellow oil. This distillate was extracted thrice with ether. The ether was distilled away and the remaining oil distilled at 135°/12 mm. This liquid was soluble in sodium hydroxide solution yielding a yellow solution. Yield: 14.5 per cent;  $D_4^{25}$ : 1.116;  $n_D^{25}$ : 1.546; m.p. phenylhydrazone, yellow in color: 109–110°.

In a second preparation the oil from the ether extract was converted directly to the Schiff's base with ethylenediamine but in another, in which the acidified reaction mass was extracted with ether and the oil obtained on evaporating the ether treated with ethylenediamine, only a pasty mass was obtained and it became necessary to work up the paste by decomposing the Schiff's base with acid, extracting with ether and steam distilling. Apparently either a steam or a vacuum distillation is necessary.

#### DI-(2-HYDROXY-3-*n*-PROPOXYBENZAL)-ETHYLENEDIIMINE

The aldehyde and diamine condensed readily and the yellow product recrystallized easily from alcohol; m.p.: 93–94°.

#### DI-(2-HYDROXY-3-*n*-PROPOXYBENZAL)-ETHYLENEDIIMINE COBALT

About 7 g. of di-(2-hydroxy-3-*n*-propoxybenzal)-ethylenediimine was dissolved in a hot solution of 40 ml. of 0.98 *N* sodium hydroxide, 30 ml. of water, 35 ml. of alcohol, and 2 g. of sodium acetate. To this was added a hot solution of 5 g. of cobalt acetate, 3 g. of acetic acid, 20 ml. of water and 10 ml. of alcohol. This provided an excess of sodium hydroxide in the first solution and enough acetic acid in the second to make the mixture definitely acidic. The cobalt acetate was in excess. A brown precipitate appeared immediately and after being boiled for fifteen minutes the solution was filtered and thoroughly washed with both alcohol and water. It was dried under a vacuum at 110° for four hours. A few particles of unactivated compound were placed on a melting point block and warmed. At about 95° the compound lost water and turned from yellowish red to brown. There was no noticeable change as the temperature was further raised to 240°. A portion of the material was heated in a vacuum at 110° for four hours and then placed in an oxygen atmosphere of 180 pounds pressure. The compound gained 3.60 per cent in weight which was lost on again heating in a vacuum. Another trial gave the same results. The theoretical oxygen-carrying capacity for the absorption of one molecule of oxygen per two cobalt atoms is 3.55 per cent. Using the *Differential Manometric Apparatus* (Paper XIII, Method C) this material was found to absorb oxygen very rapidly, being 90 per cent saturated within two minutes. An attempt was made to obtain the rate of oxygenation in air using the *Gravimetric Rate Method* (Paper XIII, Method D) but owing to the floury nature of the compound air could not be forced through the powder sufficiently rapidly. A few determinations were made on pellets of the material, 10 to 20 mesh in size, but the results were not entirely satisfactory owing to the excessively hygroscopic character of the material.



C. DI-(2-HYDROXY-3-*n*-BUTOXYBENZAL)-ETHYLENEDIIMINE  
COBALT. Co-Ox SS*o-n*-BUTOXYPHENOL

To a solution of 300 g. of pyrocatechol dissolved in 900 ml. of water, which had been previously boiled to expel air, was added 120 g. of sodium hydroxide dissolved in 300 ml. of boiled water. The reaction mixture was cooled to room temperature and 420 g. of *n*-butyl bromide (Eastman Kodak Company, No. 51) added. The mixture was refluxed for twenty hours. Upon cooling two layers separated. The upper layer, which was almost black in color, was separated and the lower layer was acidified with hydrochloric acid and extracted with ether. The ether was removed by distillation and the *o-n*-butoxyphenol so obtained added to the upper layer which had been previously separated. This liquid was then distilled and the fraction boiling 150–154°/40 mm. collected. Yield: 81 per cent.

2-HYDROXY-3-*n*-BUTOXYBENZALDEHYDE

The butoxyphenol was converted to the aldehyde by the Duff reaction; m.p.: 49°; yield: 16 per cent.

DI-(2-HYDROXY-3-*n*-BUTOXYBENZAL)-ETHYLENEDIIMINE

This Schiff's base was obtained as a yellow crystalline material by condensing the aldehyde and ethylenediamine in alcohol. It was recrystallized from alcohol; m.p.: 93–93.5°.

DI-(2-HYDROXY-3-*n*-BUTOXYBENZAL)-ETHYLENEDIIMINE COBALT

The first preparations of this compound yielded material of oxygen-carrying capacity considerably below the theoretical value. In these preparations the Schiff's base was dissolved in an aqueous solution of sodium hydroxide, a solution which was prepared only with difficulty, and treated with the cobalt salt, or a suspension of the Schiff's base in water was digested with a solution of the cobalt salt. In later preparations made in a water-alcohol medium a material was obtained which absorbed amounts of oxygen approaching the theoretical value for one molecule of oxygen per two cobalt atoms. A typical preparation was carried out in the following manner.

A quantity of 16 g. of di-(2-hydroxy-3-*n*-butoxybenzal)-ethylene-diimine was dissolved in 400 ml. of hot alcohol. To this solution was added slowly with stirring a solution containing 12 g. of cobalt acetate in 500 ml. of hot water. The compound precipitated was yellow in color and similar in appearance to the 3-methoxy compound. The mixture was digested, with occasional stirring, on the steam bath for sixty minutes, then allowed to cool before filtering on a Buchner funnel. The precipitate was reslurried in 500 ml. of hot water, again filtered and sucked as dry as possible. The material was dried at 100° either in a vacuum or in air and then activated by heating in a vacuum at 125°. Oxygen-carrying capacity: 3.32 per cent; theoretical capacity: 3.41 per cent.

RATE OF OXYGENATION OF DI-(2-HYDROXY-3-*n*-BUTOXYBENZAL)-ETHYLENEDIIMINE COBALT

The rate of oxygenation of di-(2-hydroxy-3-*n*-butoxybenzal)-ethylenediimine cobalt was determined using *The Large Volume Manometric Apparatus* described in Paper XIII (*Method G*). The data is plotted in Figure 3. The results indicate an amazing rate of oxygenation for Co-Ox

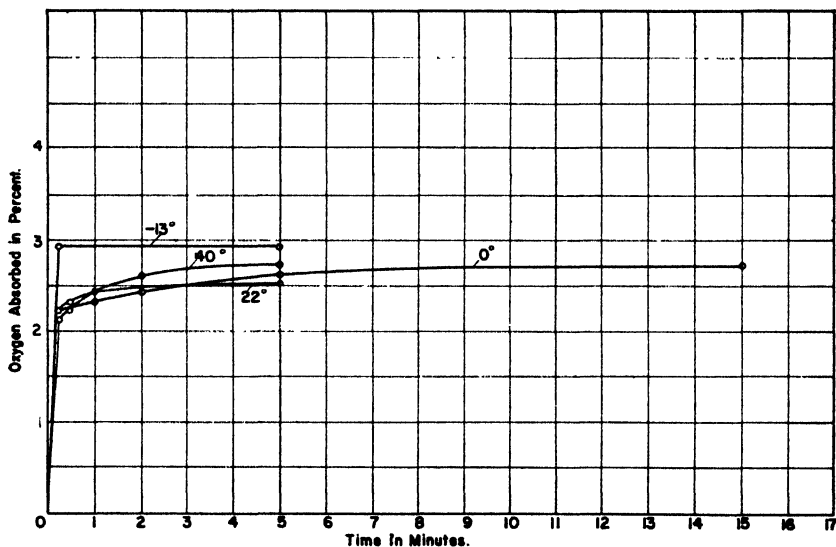


FIG. 3. Rate of oxygenation of Co-Ox SS in oxygen at various temperatures.

SS. Temperature has very little effect on the rate of oxygenation; the compound resembles 3-Ethoxy Co-Ox in this respect. The fast compounds must necessarily be those compounds whose rates of oxygenation are largely independent of temperature, the heat liberated during the oxygenation having relatively little retarding influence on the absorption of oxygen.

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[Further discussions of the "Studies on Oxygen-Carrying Cobalt Compounds" will follow in subsequent issues of the *Journal of Science*.]



# PROLONGMENT OF THE REPRODUCTIVE PHASE OF *TRYPANOSOMA LEWISI* BY THE ADMINISTRATION OF SODIUM SALICYLATE<sup>1</sup>

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The subject of two reports (1,2) from this laboratory concerned prolongment of the reproductive phase of *Trypanosoma lewisi* infection in the rat and exaltation of numbers as a result of restricting pantothenic acid in the host's diet. Our attention was attracted to sodium salicylate by the work of Ivánovics (3,4) who noted inhibition of growth of *Staphylococcus aureus* in the presence of 0.001 M of the drug, and the complete removal of the inhibition by  $10^{-7}$  M pantothenic acid, but by no other agent of biological importance. The challenge was obvious—to administer sodium salicylate to the rat host and observe how its reaction is reflected in the course of *Trypanosoma lewisi* infection. The successful result of the venture is the subject of this paper.

Before proceeding with the presentation of our work it is desirable to review two current concepts of the nature of immune reactions to *T. lewisi*. According to Taliaferro and his collaborators (5,6,18), two humoral antibodies are produced in the host during the course of the infection, both of which are associated with the globulin fraction of the serum: (1) so-called ablastin, the anti-reproduction factor that accumulates in the blood during the early days of the infection, becoming demonstrable about the fifth day and completely inhibiting reproduction by the tenth day, and (2) the trypanocidal factor, which first effects destruction of a majority of the microorganisms some time between the eighth and fourteenth day ("first number crisis") and entirely eliminates them a few days to several months later, remaining in the blood indefinitely to prevent reinfection, whose effects "are due to typical lysins which may, however, act as opsonins *in vivo*."

Augustine (7) does not accept this duality of immune bodies. From study of the behavior of the trypanosomes in reinfections of recovered rats he is led to declare that (1) the practical disappearance of dividing forms from the blood is to be explained by their low threshold sensitization to the trypanocidal antibody, which renders them selectively vulnerable to phagocytosis, and (2) the adult and some of the dividing trypanosomes are mechanically eliminated from the circulation subsequent to agglutination by the trypanocidal antibody. Thus Augustine is inclined to regard the trypanocidal antibody as the sole factor involved in immunity, although he considers the possibility of another duality, namely, an

<sup>1</sup>Supported in part by a grant from the Science Research Institute, Iowa State College.

opsonin specific for dividing trypanosomes and an agglutinin for both adult and dividing forms.

It must be remembered, however, that Augustine's observations were made entirely on reinfections, and that there are a number of serological aspects of Taliaferro's work upon which he has not touched. Since we have nothing new to contribute to the fundamental issue at this time, we shall employ such noncommittal terms as "reproductive phase" and "anti-reproductive process," meaning by the latter only the phenomenon that accounts for absence of dividing forms from the blood, whatever its ultimate nature.

Salicylates, as is well known, are widely used in medicine on account of their antipyretic, analgesic, and antiphlogistic action. There is considerable literature bearing on the physiological action of salicylates administered to mammals, but only certain of the more pertinent effects will be mentioned here. Link *et coll.* (8) produced temporary hypoprothrombinemia in rats maintained on a ration low in vitamin K with single doses of salicylic acid, but found that feeding a ration containing sufficient vitamin K, or administration of the synthetic 2-methyl-1,4-naphthoquinone, would protect against the drug dosages they tested. Minoru (9) found that small doses of sodium salicylate decreased blood sugar by parasympathetic stimulation, while large doses produced hyperglycemia by direct action on the liver, accelerating glycogen mobilization. Fashena *et coll.* (10) found that blood concentration of 350  $\mu$ g. per cc. approached the toxic. Moderate to large doses produced hypoprothrombinemia preventable by heavy dosing with vitamin K, while small doses decreased the alkali reserve.

The main pathology of salicylate poisoning, according to Troll and Menten (11), is widespread hemorrhage from capillaries in serous surfaces. Immune reactions too have been influenced by salicylates. Swift (12) found that sodium salicylate administered to rabbits impairs the formation of anti-*Streptococcus viridans* immune bodies, and Homburger (13) reported what appears to be diminished formation of anti-Rh agglutinins in guinea pigs and rabbits following injection of *Rhesus* blood cells when sodium salicylate is administered prior to and during the immunization period. Dorfman *et coll.* (14) recently noted inhibitory effects of sodium salicylate on the spreading effects of hyaluronidase *in vitro* and *in vivo*.

#### MATERIALS AND METHODS

The strain of rats and of *T. lewisi* were the same as previously reported. The rats were fed the stock ration, on which the colony is maintained, from the time of birth. Inoculation was intraperitoneal and with about 200,000 trypanosomes in 1 cc. of diluted blood. Most of the trypanosome counts were made in a Levy-Hausser haemocytometer by the red cell method, except that 20 squares were usually counted. Blood smears were stained in Wright's.

Sodium salicylate (c. p.) was dissolved in distilled water, and administered directly into the stomach through a No. 8 soft rubber

catheter attached to a glass syringe. Administration was usually started on the day of inoculation. Male rats were used except where otherwise stated, because they tolerate the salicylate much better than the females, which are likely to succumb to severe hemorrhage from the vagina.

There appears in Table 1 a column headed "D.F. %" containing percentages of division forms. The latter are to be defined as the proportion of individual trypanosomes with characteristics of size, structure, or stainability peculiar to the reproductive phase of the infection, as contrasted with the adults, which are the only forms in the blood after the anti-reproductive process is effective. Hence, Taliaferro's (5) figs. 1-7 of Fig. 1 are division forms, while figs. 8 and 9 are adults. Figs. 5-7 are transitionals, in our parlance. No measurements were made, but a specimen had to be striking in its peculiarities before it was classified with the division forms. It is realized that a degree of error ("personal factor") is inherent in the method, but it is believed that it is at least as adequate as the original Taliaferro (16) method of measuring and computing variation coefficients, for the latter does not sufficiently accent the vagaries of form encountered in a population with an accelerated tempo of reproduction. Two hundred specimens on a slide stained in Wright's were carefully scrutinized for each item in the column.

#### EXPERIMENTAL DATA

*Salicylate administration started on day of inoculation.* Table 1 records data from three experiments in which all the treated rats lived long enough for the effects of salicylate to be observed. It is to be noted, firstly, that up to and including the fifth day numbers of trypanosomes and percentage of division forms were comparable in the treated and untreated series. Secondly, these two values for the treated series greatly exceed those for the untreated series on the sixth (exp. 2) and seventh (exps. 1 and 3) days. Thirdly, the percentage of division forms is very low in the untreated rats by the eighth or ninth days, while the high values in the treated series on these days indicates the continuation of a fast tempo of reproduction. Fourthly, no reproduction is evident in the untreated series after the ninth day, while considerable numbers of division forms persisted in every surviving rat of the treated series.

Another important phenomenon brought out in the table concerns the relation between number of trypanosomes and percentage of division forms, particularly in the treated series. Following the number counts for rat 22, for example, the values are 120, 294, 350, and 390, on the fifth, seventh, and twelfth days. The D.F. percentage values of 49, 66, 73, and 77, respectively, indicate a tremendously rapid rate of reproduction that was probably more or less continuous from 48 hours after the time of inoculation till the rats became moribund on the twelfth day. It is impressive that the blood population arose from less than 100 per mm.<sup>3</sup> of blood on the inoculation date to 120,000 per mm.<sup>3</sup> on the fifth day. But during the next four days, instead of a x1200 increase it was x3. At the end of three more days the count was approximately the same, although



TABLE 1 (continued)

## Experiment 2

Day of Inf.	Treated Series (Rats 11 and 12, 0.09 g. sal. daily 2-10 to 2-24, except 2-16, 18, 21)					Untreated Series (Rats 9 and 10 received no sal.)					Remarks
	Rat No.	Wt. g.	R.B.C.	No. T.	D. F. %	Rat No.	Wt. g.	R.B.C.	No. T.	D. F. %	
0.....	11 12	159 130	760 888	.....	.....	9 10	130 146	695 752	.....	.....	Inoculated 2-10
6.....	11 12	144 138	640 498	430 380	67 73	9 10	169 162	560 869	185 30	55 43	Act. div. in both series
8.....	11 12	140 146	473 331	687 480	81 69	9 10	170 162	404 734	260 70	18 14	Do. Rat 11 died 9th day
13.....	11 12	..... 137	..... 208	..... 670	..... 16	9 10	190 172	534 804	92 25	0 0	No. act. div. in either series
15.....	12	129	384	545	3	9 10	(no counts made)				Rat 12 died 16th day



TABLE 1 (continued)

## Experiment 3

Day of Inf.	Treated Series (Rats 21, 22, and 26, 0.06 g. sal. d. 2-20 to 3-3, except 0.10 g. 2-25, 27)					Untreated Series (Rats 24 and 29 received no sal.)					Remarks
	Rat. No.	Wt. g.	R.B.C.	No. T.	D. F. %	Rat. No.	Wt. g.	R.B.C.	No. T.	D. F. %	
0.....	21 22 26	131 123 152	916 868 880	..... ..... .....	..... ..... .....	24 29	133 168	650 830	..... .....	..... .....	Inoculated 2-20
5.....	21 22 26	136 134 171	671 636 754	148 120 123	63 49 52	24 29	146 182	672 704	115 145	55 63	Act. div. in both series
7.....	21 22 26	141 135 174	430 577 648	450 294 230	65 66 59	24 29	146 188	500 555	138 180	27 33	Act. div. in both series
9.....	21 22 26	135 134 162	315 415 522	430 350 220	69 73 66	24 29	153 189	509 594	130 130	10 7	Act. div. in Tr. Ser.
12.....	21 22 26	139 166	306 287	390 320	77 67	24 29	161 198	583 598	145 100	0 0	Do. Rat 21 died 10th day; Rats 22 and 23 moribund 12th day

the percentage of division forms was about 75 throughout. Only one conclusion can be drawn: a tremendous mortality of the microorganisms attended the high rate of reproduction manifest throughout!

Other corroborative evidence of the inhibiting effect of salicylate on the anti-reproductive process was obtained while testing for the dosages optimum for striking results. The result of overdosing was death of most of the rats before the extension of the multiplicative phase could be noted, although a number of individuals did survive long enough for significant results to be obtained. An experiment that illustrates minimum borderline effective dosage is of special interest. To four rats of a litter with an average weight of 130 g. were administered 0.045 g. of salicylate daily, commencing the day of inoculation, while the other four rats were inoculated but not treated. On the ninth day actual division could not be observed in the untreated smears, though the large transitionals were seen, while it was observable in the treated smears on this and the tenth day. On the eleventh and thirteenth days the treated rats still had transitionals, but no actually dividing forms, while only adults could be found in the untreated smears. Only adults could be found in the treated smears on the fourteenth day. To summarize, the following phenomena occurred in the treated rats: (1) prolonging of the reproductive phase several days beyond the normal for this strain; (2) eventual development of the anti-reproductive process despite the administration of salicylate.

*Salicylate administration started five days or more after inoculation.* Two lots of four female rats were subjected to separate experiments to determine whether salicylate would inhibit the anti-reproductive process when administration is delayed for five full days. It was mentioned above that ablastin is demonstrable in the blood of the normal rat five days after inoculation. Lot 1 was composed of young rats averaging 141 g.; Lot 2 of old rats, all of which had had litters and failed to breed further, averaging 220 g. Just 120 hours after inoculation each rat of Lot 1 was injected directly into the stomach with 0.09 g. sodium salicylate in 2 cc. of distilled water; Lot 2 with 0.135 g. of the chemical in the same dilution. There were actively dividing trypanosomes in the blood of all the rats at this time. The plan was to inject the rats of Lot 1 with 0.06 g. of the drug on the sixth, seventh, ninth, eleventh, fourteenth, and seventeenth days, but only one rat in the lot survived the entire course of treatment. The plan for Lot 2 was the same except that the drug dosage was 1.5x as large. Only one rat in this lot survived. Dividing trypanosomes were demonstrable in the survivor of Lot 1 on each day the drug was administered and on the eighteenth day, when the rat was sacrificed on account of its moribund condition.

A more detailed study was made of rat 64, the survivor of Lot 2. This female rat weighed 228 g. on the inoculation date, and 226 g. on the fifth day of the infection, the time of the first administration of salicylate.

Since division forms could no longer be found upon casual inspection of smears from the single control on the ninth day, we then commenced

to enumerate the adults, forms notably smaller than adults, forms notably larger than adults ("transitionals"), and actually dividing forms of whatever type in the blood of rat 64. The percentages of each on the different dates are shown in Table 2. Although no actually dividing

TABLE 2  
DAY-BY-DAY FINDINGS (PERCENTAGES)

	9th day	10th day	11th day	13th day	15th day	17th day	19th day
Adults . . . . .	88.5	83	61.5	72	81	86	90
Small . . . . .	6.5	4	10	5.5	6.5	6	3
Large . . . . .	3.5	10.5	26.5	21.5	11	8	7
Dividing . . . . .	1.5	2.5	2	1.0	1.5	0	0
Rat weight . . . .	219 g.	215 g.			206 g.	176 g.	168 g.

specimens were observed among the 200 classified on each of the seventeenth and nineteenth days, they could readily be found elsewhere in the smears. The rat was killed on the nineteenth day because it was moribund.

Two similar experiments involving eight male rats were performed in which the salicylate (same dosage as above) was started at the end of the ninth day. No actual dividing forms were observed at this time, but a few to many of the transitional forms, i. e., the large, thick, basophilic, nondividing types, were encountered in each smear. At no time could dividing forms be found in the stained smears after salicylate administration started. An almost identical experiment was performed with two male rats six days along in the infection, with identical results. Since no actually dividing forms could be located in any of the ten rats involved in these experiments, it seems to be definitely established that the salicylate is not effective unless considerable numbers of them are still in the blood.

*Salicylate administration started after trypanosome population becomes adult, or after recovery.* Following are notes from the protocols of experiments performed for the purpose of reinitiating the reproductive phase after all division forms are absent from the blood, or after recovery:

1. Rats 1A ♂ and 2A ♀. Inoculated December 4; weights 95 g. and 82 g., respectively. Heavy populations of adult trypanosomes persisting on February 7; weights 186 g. and 183 g.; daily dosing with 0.08 g. sodium salicylate begun. Dosage increased to 0.12 g. on February 15. No division forms seen in stained blood smears made every other day to deaths on February 27 and 24, respectively.

2. Rats 3A ♂, 4A ♂, and 5A ♀. Inoculated January 14; weights 120 g., 127 g., and 113 g., respectively. No division forms appearing on tenth day, when daily dosing with 0.08 g. salicylate was begun. Examinations of stained blood smears on alternate days to February 25 revealed no dividing trypanosomes.

3. Rats 6A ♂, 7A ♂, and 8A ♀. Inoculated January 22; weights 121 g., 148g., and 132 g., respectively. Trypanosome counts on tenth day were

88,000, 110,000, and 42,000, respectively, per mm.<sup>3</sup> of blood; daily dosing with 0.08 g. salicylate begun. Stained blood smears made on alternate days to February 26 revealed no division forms.

4. Rats 9A ♂, 10A ♀, 11A ♂, and 12A ♂. Inoculated December 4; average weight 168 g.; infection ensued; blood cleared of trypanosomes by February 11. Commencing the latter date each rat received 0.08 g. salicylate daily. Inoculated i. p. with trypanosomes on February 12, 18, 23, 28 (when rat 10A succumbed). Blood was negative for trypanosomes to March 4. Treatment discontinued till March 11, when each survivor, still negative, was administered 0.25 g. salicylate through a stomach tube and 200 million washed trypanosomes via the jugular vein. Tail blood swarmed with trypanosomes immediately after injection, but was negative the next morning. Thus the administration of salicylate had not broken down the host's immunity to reinfection. If the Taliaferroan doctrine is correct, the destruction of ablastin would have been masked by the effect of the trypanocidal antibody anyhow, unless it too had been destroyed.

#### DISCUSSION

The administration of sodium salicylate at the stated levels and intervals resulted in failure of the immune reaction normally manifested by the disappearance of dividing *Trypanosoma lewisi* from the blood between the sixth and tenth days. Success in inhibiting the anti-reproductive process was attained also when salicylate was withheld until the end of the fifth day, when dividing forms were still numerous in the blood. Administrations begun at the end of either the sixth or ninth days, when numerous adult trypanosomes and considerable numbers of transitionals were present in the smears, were without success in reinitiating a reproductive phase. Likewise, administrations of salicylate to recovered rats did not make for reinfection following reinoculation with adult trypanosomes.

The complete interpretation of these phenomena is manifestly impossible, though it is clear that the course of the infection was altered by prolonging its reproductive phase and that an exaltation of the parasitemia resulted. These results were comparable to those previously obtained from pantothenic acid deficiency. The similarity of results in this and the previous investigation suggest, but of course do not prove, that salicylate may interfere with the utilization of pantothenic acid in the immune reaction.

It is attractive to think of the antibody responsible for the anti-reproductive process as an oxidation enzyme. According to Werkman and Wood (17) such an enzyme (or holoenzyme) would consist of a protein (apoenzyme) united to a coenzyme or prosthetic group. The protein moiety is ordinarily highly specific. The coenzyme is usually an organic compound, although the term has been applied to inorganic ions also. Taliaferro (18) states that the reaction product which inhibits the reproduction of *T. lewisi* is a protein, or very closely associated with a protein

of the serum globulin. The previously cited work has shown that the reproductive phase of *T. lewisi* infection may be prolonged by withholding pantothenate from the rat's diet, and it is shown in the present paper that administration of sodium salicylate has the same effect.

For these reasons we are suggesting as a working hypothesis that the antibody which finds expression in the anti-reproductive process is an oxidation enzyme, composed of a protein moiety in union with pantothenic acid. (The suggestion is made with the full realization of the possibility that the observed effects might eventually be shown to be due to some other process, such as protein denaturization or interference with vitamin K utilization.) The apoenzyme would be a specific protein that accumulates in the rat's blood during the first nine days of the infection, becoming associated with the coenzyme, pantothenic acid, which is normally present. This hypothesis would account for failure or partial failure of the anti-reproductive process in rats on diets deficient in pantothenate. The blocking of the process with salicylate could be explained by this drug masquerading as coenzyme and uniting with the specific protein to the practical exclusion of pantothenate, and forming a reaction product other than the holoenzyme. The preemptory activity of the salicylate occurs when the host is on a normal diet, or even when a considerable amount of supplementary pantothenate is fed (unpublished experiments).

Euler (19) has reported that sodium salicylate weakens the affinity of apozymase for cozymase. A similar explanation might be applicable here also.

Failure to break down the anti-reproductive principle (holoenzyme), once it is formed, with heavy administrations of salicylate is of no special weight against the hypothesis, for it is well known that in certain cases disassociation of the coenzyme from the protein does not readily occur with present methods (V. Werkman and Wood, 17).

It is unfortunate that our results with salicylate have little bearing on the nature of the anti-reproductive process. The observed phenomena, however, are not in disagreement with the Taliaferroan doctrine of the existence of ablastin, which inhibits reproduction. In this light it could be claimed that salicylate prevents the formation of this reaction product when heavy doses are administered, and that its formation is slowed down when dosages are lighter. Salicylate so far has not affected ablastin or trypanolysin, once formed. It is evident from the successive trypanosome counts for individual rats together with the high percentage of division forms, appearing in Table 1, that terrific destruction of trypanosomes (trypanocidal antibody in action?) was occurring in the treated series in the face of heavy multiplication and only mild rise in numbers. How the Augustinian doctrine of special selectivity of a single trypanocidal body for division forms could account for the phenomenon is not clear.

#### CONCLUSIONS

1. Prolonging of the reproductive phase of the *Trypanosoma lewisi* infection and exalted parasitemia may result from the repeated admin-

istration of sodium salicylate begun on the date of inoculation or at the end of the fifth day.

2. Administration of salicylate commenced at the end of the sixth or ninth days, when dividing forms could no longer be located in the blood smears with the microscope, did not result in reinitiation of the reproductive phase.

3. Recovered rats could not be reinfected following a course of salicylate.

4. The suggestion is advanced that the antibody which is responsible for the anti-reproductive process may be in the nature of an oxidation enzyme, one moiety of which is pantothenic acid.

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# FLORA OF ALASKA AND ADJACENT PARTS OF CANADA<sup>1</sup>

An Illustrated and Descriptive Text of All Vascular Plants Known  
to Occur Within the Region Covered

## PART VI. CRASSULACEAE TO FABACEAE

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### 18. CRASSULACEAE (Stone-crop Family)

Mostly fleshy or succulent herbs; flowers regular, borne in cymes; sepals, petals, and carpels each 4 or 5, with stamens of the same number or twice as many; carpels distinct or nearly so with a small scale at the base of each; fruit composed of dry, dehiscent follicles.

#### SEDUM L.

Fleshy herbs with flowers borne in terminal, often one-sided cymes; leaves alternate, often imbricate; sepals distinct or somewhat united; stamens 8–10, the alternate ones usually attached to the petals; carpels 4 or 5, distinct or united at the base. (Latin, to sit, in allusion to the habit of the plants).

1A. Petals united below ..... 1. *S. oregonum*

2A. Petals distinct.

1B. Flowers polygamous or dioecious, leaves broad ..... 2. *S. roseum*

2B. Flowers perfect, leaves terete ..... 3. *S. stenopetalum*

1. *S. oregonum* Nutt.

Oregon Sedum

*Gormania oregona* (Nutt.) Britt.

Rootstock rather slender, creeping; stems erect or ascending, often curved, 6–15 cm. tall; leaves spatulate-cuneate, glabrous, 8–20 mm. long; cymes rather congested, with a leafy involucre; calyx lobes lanceolate, about 4 mm. long; petals narrowly lanceolate, acuminate, 10–12 mm. long, united about one fourth their length, yellow often tinged rose.

Southeastern Alaska—northern California. Fig. 583.

2. *S. roseum* (L.) Scop.

Roseroot. Rosewort

*Rhodiola rosea* L.

Rootstock thick, fleshy or woody, rose-scented; stems leafy, somewhat glaucous, 1–3 dm. tall; leaves oblanceolate or obovate, entire or dentate, 1–4 cm. long, the lower ones smaller; petals in the type form yellow, in the ssp. *integrifolium* (Raf.) Hult. (*Rhodiola integrifolia* Raf.) dark reddish purple; follicles erect with widely divergent tips. Var.

<sup>1</sup>Preceding parts of the paper were published in this Journal as follows: Part 1, Vol. XVIII, pp. 137–175, 1943; Part 2, Vol. XVIII, pp. 381–446, 1944; Part 3, Vol. XIX, pp. 133–205, 1946; Part 4, Vol. XX, pp. 213–257, 1946; Part 5, Vol. XX, pp. 297–347, 1946.



*frigidum* (Rydb.) Hult. (*Rhodiola alaskana* Rose) averages taller, leaves sharply toothed in upper third, drying thin.

The type form has been collected at Nome, ssp. *integrifolia* is common in most of our area, var. *frigidum* occurs in the Pacific Coast and Bering Sea regions, the species being circumboreal. Fig. 584.

3. *S. stenopetalum* Pursh.

Narrow-petaled Sedum

Tufted perennial; rootstock slender, branching; stems 7–18 cm. tall; leaves linear, sessile, 5–15 mm. long, imbricate on the sterile shoots; cyme 3- to 7-forked; pedicels short; calyx lobes lanceolate; petals yellow, narrowly lanceolate, acuminate, 6–7 mm. long; follicles about 4 mm. long, the tips divergent.

S. Yukon—Sask.—Nebr.—northeastern Calif. Fig. 585.

19. SAXIFRAGACEAE (Saxifrage Family)

Ours all herbs, mostly perennial; leaves alternate, rarely opposite, or often all basal, usually without stipules; flowers in ours perfect and regular or nearly so; hypanthium often more or less adnate to the ovary; sepals and petals 5 or rarely 4 or the petals wanting; stamens as many or twice as many as the sepals except in *Tolmiea* which has only 3; carpels 1—several, usually 2, distinct or united; fruit a capsule or composed of follicles.

- 1A. Staminoidea present between the stamens ..... 1. *Parnassia*
- 2A. Staminoidea not present.
  - 1B. Petals none, low herbs ..... 2. *Chrysosplenium*
  - 2B. Petals usually present.
    - 1C. Petals fringed or laciniate-lobed.
      - 1D. Calyx flat at base ..... 3. *Mitella*
      - 2D. Calyx cup-shaped at base ..... 4. *Tellima*
    - 2C. Petals entire.
      - 1D. Stamens 3 ..... 5. *Tolmiea*
      - 2D. Stamens 5.
        - 1E. Capsule 1-celled, leaves mostly basal ..... 6. *Heuchera*
        - 2E. Capsule 2-celled, stem leafy ..... 7. *Boykinia*
      - 3D. Stamens 8 or 10.
        - 1E. Carpels unequal ..... 8. *Tiarella*
        - 2E. Carpels equal.
          - 1F. Leaves leathery, carpels nearly distinct ..... 9. *Leptarrhena*
          - 2F. Leaves not leathery, carpels more or less united ..... 10. *Saxifraga*

1. PARNASSIA L.

Glabrous, scapose perennials; leaves basal, petioled, entire; flowers perfect, solitary, terminal, white or yellowish; scapes usually bearing 1 leaf; sepals and petals each 5; stamens 5, alternating with the petals and with 5 clusters of gland-tipped staminoidea; carpels 3 or 4, united; ovary with 3 or 4 parietal placentae; fruit a 1-celled loculicidal capsule. (Name from Mt. Parnassus in Greece.)

- 1A. Petals fimbriate on the sides ..... 1. *P. fimbriata*
- 2A. Petals entire.
  - 1B. Petals scarcely equaling the sepals, 3-veined ..... 4. *P. kotzebuei*

2B. Petals longer than the sepals, 5- to 9-nerved.

1C. Petals nearly twice as long as the sepals,  
staminoidea, 8-15 in each fascicle..... 2. *P. palustris*

2C. Petals only slightly exceeding the sepals,  
staminoidea 7-9 in each fascicle..... 3. *P. montanensis*

1. *P. fimbriata* Konig.

Fringed Grass-of-Parnassus

Leaf-blades reniform to cordate, 2-4 cm. wide; scapes 2-5 dm. tall, with 1 sessile leaf above the middle; sepals elliptical, obtuse, about 5 mm. long; petals nearly twice as long as the sepals, obovate; staminoidea 5-9 in each fascicle.

Central Pacific coast of Alaska—Yukon—Utah—N. Mex.—Calif. Fig. 586.

2. *P. palustris* L.

Northern Grass-of-Parnassus

Leaves cordate, 1-3 cm. wide; scapes 1-5 dm. tall, bearing a cordate-clasping leaf below the middle; sepals ovate-lanceolate, strongly veined, 5-7 mm. long; petals oval, 8-12 mm. long; capsule ovoid, about 1 cm. long. The inland race has more deltoid stem leaves, narrower sepals and broader-clawed staminoidea than the type form and has been separated as var. *neogaea* Fern.

Common in wet places, circumboreal. Fig. 587.

3. *P. montanensis* Fern. & Rydb.

Montana Grass-of-Parnassus

Leaves ovate with subcordate or rounded base, 10-20 × 8-18 mm.; scapes about 2 dm. tall, the leaf ample, ovate, borne below the middle; sepals acute, 7- to 9-veined, 7-9 mm. long; capsule round-ovoid, about 1 cm. long.

Yukon—Sask.—Mont.

4. *P. kotzebuei* C. & S.

Kotzebue Grass-of-Parnassus

Leaves ovate, narrowed, truncate or subcordate at the base, 1-2 cm. long; scapes naked or with a leaf near the base, 6-15 cm. tall; sepals oblong-lanceolate, 5-6 mm. long, about the same length as the petals; staminoidea 3-5 in each fascicle.

East Asia—Coronation Gulf—Labr.—Greenl.—Newf.—Wyo. Fig. 588.

*P. parviflora* DC. has been reported from Alaska but the reports need confirmation. It has leaves with acute bases, the stem-leaf at or a little below the middle, petals about the same length as the sepals, and staminoidea 5-7 in each fascicle.

2. CHRYSOSPLENIUM (Tourn.) L.

Low, glabrous, somewhat succulent herbs usually growing in very wet places; leaves petioled, crenate; flowers axillary or terminal; hypanthium adnate to the lower portion of the ovary; sepals usually 4; petals none; capsule 1-celled with 2 parietal placentae, many seeded; seed smooth, shining. (Greek, golden spleen, from reputed medicinal virtues.)

Stamens 4..... 1. *C. tetrandrum*  
Stamens 8..... 2. *C. wrightii*

1. *C. tetrandrum* Th. Fries. Northern Water Carpet

A stoloniferous perennial; stems 3–15 cm. tall, bearing several leaves; leaf blades reniform or orbicular with 3–5, rarely 7 rounded teeth, truncate to cordate at the base, 4–12 mm. wide; sepals usually 4; stamens opposite the sepals; seeds several, brownish red.

Wet places, circumpolar. Fig. 589.

2. *C. wrightii* Franch. & Sav. Bering Sea Water Carpet  
*C. beringianum* Rose

Perennial with a rather thick, scaly rootstock; leaves thick, coriaceous, 3- to 7-lobed with rounded divisions, the petioles usually with brownish hairs; flowering stem short and stout, almost leafless except at apex, many-flowered, the flowers clustered; sepals short and broad, rounded.

E. Asia—Yukon—Aleutian Isls. Fig. 590.

## 3. MITELLA (Tourn.) L.

Perennials; leaves cordate, orbicular, or ovate, clustered on a scaly rootstock; stems scape-like, naked or with a few leaves; inflorescence a simple raceme; hypanthium saucer-shaped, adnate to the ovary; flowers white or greenish; petals 5, pectinately pinnatifid; filaments short; ovary 1-celled with 2 parietal or almost basal placentae; styles 2, very short. (Diminutive of Mitra, a cap.)

Stamens 5..... 1. *M. pentandra*  
Stamens 10..... 2. *M. nuda*

1. *M. pentandra* Hook. Alpine Mitrewort  
*Pectianthia pentandra* (Hook.) Rydb.

Leaves cordate, crenate, indistinctly lobed, 2–5 cm. wide; flowering stem naked or with 1 small leaf, 1–3 dm. tall, hirsute, glandular above; racemes lax, the flowers often in pairs; sepals broadly triangular; petals cut into 5–9 capillary pinnae; stamens with very short incurved filaments and reniform anthers.

Wet alpine meadows, Kodiak—southeastern Alaska—Colo.—northern Calif. Fig. 591.

2. *M. nuda* L. Stoloniferous Mitrewort

Stoloniferous; flowering stems usually naked, pubescent, 5–20 cm. tall; leaves reniform-orbicular, cordate at the base, crenate or doubly crenate, 12–40 mm. wide, pubescent with scattered hairs; flowers few, greenish; petals pinnately divided into filiform segments; filaments subulate, more than half as long as the sepals.

Southern Yukon—Newf.—N. S.—Penn. Fig. 592.

## 4. TELLIMA R. Br.

Hirsute perennial; rootstock thick and scaly; leaves palmately lobed, parted or divided; flowers in an elongated raceme on a scape-like stem; sepals ovate, erect; petals white, purplish, or yellowish, spreading or

reflexed, pinnately laciniate; stamens 10, included; carpels 2, ovary 1-celled with 2 many-ovuled parietal placentae; capsule 2-valved, adherent to the base of the hypanthium. (An anagram of *Mitella*.)

*T. grandiflora* (Pursh) Dougl.

Fringe Cup

Leaves cordate or reniform, sparingly hirsute on both sides, shallowly lobed, dentate, 4–10 cm. wide; flowering stems 3–10 dm. long, hirsute with long hairs, bearing 2 or 3 leaves; inflorescence 1–3 dm. long, glandular; hypanthium cup-shaped, about 8 mm. long.

Unimak Isl.—southeastern Alaska—Selkirk Mts.—northern Calif. Fig. 593.

#### 5. TOLMIEA Torr. & Gray

Perennial with a scaly caudex; leaves many, mostly basal, with stipules; flowers borne in long terminal racemes; sepals united into a long tube split on one side; petals filiform; ovary 1-celled, stipitate, with 2 equal carpels and parietal placentae. (Dr. W. F. Tolmie was a collector and surgeon of the Hudson Bay Co.)

*Tolmiea menziesii* (Pursh) Torr. & Gray

Youth-on-Age

*Leptaxis menziesii* (Pursh) Raf.

Basal leaves cordate, acute, cuspidate-toothed, hirsute, ciliate, 2–12 cm. wide, on long petioles; stems up to 1 m. tall with a few—several leaves; flowers on slender pedicels subtended by small fimbriate bracts; petals capillary, brown, exerted from the sinuses between the sepals; fruit protruding through the slit on the lower side of the hypanthium. Propagates vegetatively by new plants forming in the sinuses of the leaves.

Southeastern Alaska—Calif. Fig. 594.

#### 6. HEUCHERA L.

Perennials with thick, scaly rootstocks; leaves mostly radical, long-petioled; stems scape-like, bearing racemes or panicles of small whitish or purplish flowers; hypanthium adherent to lower portion of ovary, often oblique; sepals 5, often unequal; petals 5, small; ovary 1-celled, with 2 parietal, many-ovuled placentae; styles 2, slender. (Johann Heinrich von Heucher was a German botanist.)

*H. glabra* Willd.

Alpine Heuchera

Basal leaves cordate, 5- to 7-lobed, thin and shining, doubly serrate, 4–12 cm. long; flowering stems 2–6 dm. tall, 1- to 3-leaved; panicle lax; sepals ovate, scarcely 1 mm. long; petals ovate, clawed, about twice as long as the sepals.

Bering Sea—central Alaska—Selkirk Mts.—Ore. Fig. 595.

#### 7. BOYKINIA Nutt.

Glandular-pubescent perennials with thick, scaly rootstocks; leaves alternate, petioled; flowers in terminal panicles; hypanthium adnate to lower half of ovary; sepals 5, lanceolate or ovate-lanceolate, petals 5,

whitish; filaments short; ovary and capsule 2-celled with axial placentae; seed numerous, shining, punctate. (Dr. Boykin was a physician of Georgia.)

*B. richardsonii* (Hook.) Gray

Richardson Saxifrage

*Therefon richardsonii* (Hook.) O.Kze.

Plant with large glands raised on thick pedicels, 3–10 dm. tall; leaves mostly basal with long petioles and blades reniform to orbicular in outline, shallowly lobed and doubly toothed, the margins with prominent glands, deeply cordate at the base, 5–15 cm. wide; stem leaves reduced; hypanthium campanulate, about 5 mm. long; sepals triangular-ovate, 4–5 mm. long; petals about 1 cm. long.

Bering Str.—Arctic coast—Yukon—central Alaska. Fig. 596.

### 8. TIARELLA L.

Perennials with scaly rootstocks; leaves mostly basal, petioled, with small stipules adnate to the base; stems erect, the flowers small, white; hypanthium short-campanulate, nearly free from the ovary; sepals 5, ovate or lanceolate, petals 5, clawed or filiform; stamens exerted; carpels 2, very uneven in fruit, membranous; seed few, smooth. (Diminutive of tiara, from the form of the capsule).

Leaves trifoliolate..... 1. *T. trifoliata*  
Leaves not divided..... 2. *T. unifoliata*

#### 1. *T. trifoliata* L.

Trifoliolate Foamflower

Leaves and upper part of petiole hirsute; leaflets ovate to rhomboid, slightly lobed and with mucronate teeth, 2–9 cm. long; flowering stems 15–50 cm. tall, 1- to 3-leaved, glabrate below, glandular-pubescent above; inflorescence a narrow panicle; sepals whitish, scarcely 2 mm. long; petals very narrow; valves of capsule in fruit 4–5 and 7–9 mm. long.

Unga Isl.—southeastern Alaska—Ore. Fig. 597.

#### 2. *T. unifoliata* Hook.

Unifoliolate Foamflower

Similar to *T. trifoliata* but the leaves broadly cordate, 3- to 5-lobed, 4–10 cm. wide; lower carpel of fruit twice as wide as the upper one.

Southeastern Alaska—western Alta.—western Mont.—Calif. Fig. 598.

### 9. LEPTARRHENA R. Br.

Perennial with horizontal rootstock; leaves thick, leathery, crowded at the base of the scape; flowers small in a terminal panicle; hypanthium flattened; sepals 5, erect; petals 5, white, persistent; filaments subulate; carpels 2, united at the base, the tips slightly divergent in fruit. (Greek, delicate and male, probably referring to the slender stamens.)

#### *L. pyrolifolia* (D. Don) Ser.

Leather-leaf Saxifrage

Leaves ovate to obovate, glabrous, deep green and shining above, pale beneath, obtuse, serrate, narrowed into a short petiole, the blade 3–12 cm. long; scape with 2 reduced and clasping leaves, glabrous below,

glandular-pubescent above; sepals ovate, about 1.5 mm. long; petals narrow, longer than the sepals; follicles 6–8 mm. long.

Wet places, Aleutians—southeastern Alaska—western Mont.—Wash. Fig. 599.

# 10. SAXIFRAGA (Tourn.) L.

Perennials with perfect flowers; hypanthium free or adnate to the base of the usually 2-celled ovary; sepals and petals each 5; stamens 10; styles short; ovules numerous on axial placentae; Capsule 2-beaked (except in one or two species), many seeded; seed small. (Latin, rock and to break, referring to the habitat of many of the species.)

- 1A. Leaves opposite, plants matted.....29. *S. oppositifolia*
- 2A. Leaves alternate or basal.
- 1B. Leaves entire, not toothed.
- 1C. Flowers not or very slightly rising above the leaves.
- 1D. Margins of the leaves ciliate.....26. *S. eschscholtzii*
- 2D. Leaves glabrous, not ciliate.....19. *S. aleutica*
- 2C. Flowers on elongated stems.
- 1D. Stems low, scapose, with 1 or 2 leaves.
- 1E. Flowers yellow.....20. *S. serpyllifolia*
- 2E. Flowers white.....23. *S. tolmiei*
- 2D. Stems taller with several leaves.
- 1E. Leaves glabrous, not ciliated.....21. *S. hirculus*
- 2E. Leaves ciliated or pubescent.
- 1F. Plants with long flagelliform stolons.....22. *S. flagellaris*
- 2F. Plants without stolons.
- 1G. Leaves glabrous with ciliated margins.....24. *S. bronchialis*
- 2G. Leaves glandular-pubescent.
- 1H. Stems 2–8 cm. tall.....5. *S. adscendens*
- 2H. Stems 2–4 dm. tall.....16. *S. integrifolia*
- 2B. Leaves toothed or lobed.
- 1C. Basal leaves orbicular or reniform as broad as long (see also 13. *S. lyallii*).
- 1D. Flowering stems scape-like.
- 1E. Leaves with 3-toothed lobes.....28. *S. mertensiana*
- 2E. Leaves simply toothed.
- 1F. Flowers in a narrow spike-like panicle.....12. *S. spicata*
- 2F. Flowers in a head-like or corymb-like panicle.
- 1G. Leaves small, 10–15 mm. wide.....27. *S. nudicaulis*
- 2G. Leaves larger.....14. *S. punctata*
- 2D. Flowering stems leafy.
- 1E. Plants with bulblets, only terminal flower developing.....3. *S. cernua*
- 2E. Lateral flowers developed.
- 1F. Petals about 1 cm. long.....4. *S. radiata*
- 2F. Petals shorter.
- 1G. Plants stout, leaves 5- to 8-lobed.....1. *S. bracteata*
- 2G. Plants slender, leaves 3- to 5-lobed.....2. *S. rivularis*
- 2C. Basal leaves longer than broad (except sometimes in *S. lyallii*).
- 1D. Leaves 3- to 5-lobed.....6. *S. caespitosa*
- 2D. Leaves not lobed.
- 1E. Basal leaves cuneate-oblong or cuneate-oblancheolate.
- 1F. Leaves stiff with 3 acute teeth at apex.....25. *S. tricuspidata*
- 2F. Leaves not stiff with 3 rounded teeth at apex..5. *S. adscendens*
- 3F. Leaves with several teeth, bulblets usually present.
- 1G. Inflorescence with long ascending branches..18. *S. ferruginea*
- 2G. Inflorescence with short, rigid branches.....17. *S. foliolosa*
- 2E. Basal leaves flabellate or cuneate-obovate.
- 1F. Filaments clavate, broadest at middle.....13. *S. lyallii*
- 2F. Filaments subulate.
- 1G. Branches of inflorescence short and thick...15. *S. unalaschensis*

- 2G. Branches of inflorescence longer and thinner..... 10. *S. davurica*  
 3E. Basal leaves ovate or oval.  
 1F. Flowers in a spike-like panicle..... 7. *S. hieracifolia*  
 2F. Flowers paniculate or in terminal cluster.  
 1G. Leaves glabrous on both surfaces..... 8. *S. nivalis*  
 2G. Leaves with reddish-brown pubescence on lower surface..... 9. *S. rufidula*  
 3G. Leaves pubescent on both surfaces..... 11. *S. reflexa*

1. *S. bracteata* D. Don. Bracted Saxifrage

Stems often tufted, pubescent, at least above, 3–20 cm. tall; leaf blades reniform or orbicular, 1–4 cm. wide, mostly 3- to 7-lobed, those of the upper part of stem often 3-lobed and nearly sessile, the basal on long petioles, cuneate to cordate at the base with bulblets at the base of the petiole; inflorescence rather congested; hypanthium 3–4 mm. long; sepals ovate, 3–4 mm. long; petals 5–6 mm. long; fruit 7–8 mm. long.

East Asia—Bering Str. district—Kodiak Isl. Fig. 600.

2. *S. rivularis* L. Alpine Brook Saxifrage

Stems usually tufted, 1- to 3-flowered, glabrous or finely glandular-pubescent, 3–9 cm. tall; leaves fan-shaped or reniform, 3- to 5-lobed, those of the stem sometimes entire, 3–10 mm. wide; sepals ovate, about 2 mm. long, obtuse; petals white or purplish, nearly twice as long as the sepals; tips of the fruiting carpels widely divergent.

Wet alpine situations, circumpolar. Fig. 601.

3. *S. cernua* L. Nodding Saxifrage

Stems slender, ascending, pubescent, 8–25 cm. tall, with bulblets at the base; basal and lower stem leaves petioled, reniform, 5- to 7-lobed, 6–25 mm. wide; upper stem leaves sessile, 3-lobed or entire, bearing bulblets in the axils; flower often nodding; sepals about 3 mm. long; petals 6–9 mm. long; fruit seldom developing.

Alpine and circumpolar. Fig. 602.

4. *S. radiata* Small.

Stems more or less glandular-pubescent, 7–20 cm. tall; basal and lower stem leaves reniform or orbicular-flabelliform, petioled, 5- to 7-lobed, 10–22 mm. wide; uppermost stem leaves simple; flowers 2–7, none replaced by bulblets; sepals 2–3 mm. long; petals 8–13 mm. long; fruiting carpels 7–8 mm. long.

Bering Sea region of Asia and Alaska—Herschel Isl.—central Yukon. Fig. 603.

5. *S. adscendens* L. ssp. *oregonensis* (Raf.) Bacigalupi.

Wedge-leaved Saxifrage

Plants tufted, glandular-pubescent, 2–8 cm. tall; basal leaves imbricated, pubescent, oblong-spatulate, entire or with 3 rounded teeth at apex, 5–15 mm. long; stem leaves often purplish; sepals about 2 mm. long; petals white, 3–5 mm. long.

Rare in Alaska—B.C.—Ore. Fig. 604.

6. *S. caespitosa* L. ssp. *sileneflora* (Sternb.) Hult. Tufted Saxifrage  
*Muscaria sileneflora* (Sternb.) Small.

Densely tufted, glandular-pubescent, with leaves crowded on the caudices; leaves 8–18 mm. long, fan-shaped, 3- to 5-lobed at apex, the lobes lanceolate to linear; scapes 5–15 cm. tall, 1- to 3-flowered, bearing 2 or 3 reduced leaves; hypanthium campanulate; sepals ovate, 2–3 mm. long; petals white, 4–6 mm. long; fruit 7–10 mm. long.

This species is circumboreal. Fig. 605.

7. *S. hieracifolia* Wallst. & Kit. Hawkweed-leaved Saxifrage

Leaves ovate, narrowed into margined petioles, usually acute, the margins ciliate, toothed, 3–7 cm. long; scapes 1–5 dm. tall, glandular-pubescent; inflorescence resembling a bracted, interrupted spike, the flowers densely gregarious in the axils of the bracts; sepals triangular-ovate, 2–3 mm. long; petals purple, narrow, about as long as the sepals; fruit purplish, 5–6 mm. long. Var. *rufopilosa* Hult. has reddish-brown hairs on the under surface of the leaves.

Distribution interrupted circumpolar. Fig. 606.

8. *S. nivalis* L. Alpine Saxifrage

Leaves ovate, crenate-serrate, cuneate at the base, rounded at the apex, 1–4 cm. long, purplish beneath; scapes 4–16 cm. tall, several- to many-flowered, glandular-pubescent, especially in the inflorescence; sepals ovate-triangular, 1.5–2 mm. long; petals white, about 3 mm. long; carpels in fruit purplish, about 5 mm. long, the tips divergent.

Circumpolar. Fig. 607.

9. *S. rufidula* (Small) Engl. & Irmscher. Rusty Saxifrage

Leaves similar to those of *S. nivalis* or *S. reflexa* but bright green and glabrous or essentially so on the upper surface and densely red-tomentose beneath; scapes 5–20 cm. tall, somewhat purplish, pubescent below, inconspicuously so or glabrate above; sepals glabrous, 2–2.25 mm. long; petals white with short claw.

Has been reported from southeastern Alaska—B. C.—Ore.

10. *S. davurica* Willd. ssp. *grandipetala* (Engl. & Irmscher) Hult.

Leaves ascending, the blades flabellate, cuneate at the base, coarsely several-toothed above, glabrous or nearly so, 1–3 cm. long; scapes 6–16 cm. tall, somewhat glandular-pubescent; flowers few-several; sepals 1.5–2 mm. long, purple, reflexed; petals white, up to 5 mm. long; mature carpels erect, 6–8 mm. long.

Eastern Asia—central Alaska. Fig. 608.

11. *S. reflexa* Hook. Yukon Saxifrage  
*Micranthes yukonensis* Small.

Leaves ovate, coarsely crenate, more or less hirsute or pubescent on both surfaces, 15–50 mm. long; scapes usually more than one, 8–45 cm. tall, glandular-pubescent, many-flowered; sepals 2–2.5 mm. long; filaments



dilated in upper portion; fruiting carpels 3–5 mm. long, the tips divergent.  
Dry situations, Bering Sea—Northwest Territories. Fig. 609.

12. *S. spicata* D. Don.

Spiked Saxifrage

*Micranthes galacifolia* Small.

Leaves ascending, the blades reniform to oval, 3–9 cm. wide, crenate-dentate with gland-tipped teeth, cordate at base, with petioles 4–18 cm. long; scapes 18–65 cm. tall, glandular-pubescent, the inflorescence spike-like; sepals about 2 mm. long, reflexed; petals yellowish, about 4 mm. long; fruiting carpels 6–10 mm. long.

Bering Str.—Yukon—southwestern Alaska. Fig. 610.

13. *S. lyallii* Engler.

Red-stemmed Saxifrage

Glabrous below, usually glandular in the inflorescence, 8–30 cm. tall; leaves fan-shaped, rounded at the apex, cuneate at the base, regularly serrate on the rounded portion, 1–4 cm. wide; scapes several- to many-flowered; sepals ovate, acute, reflexed, about 2.5 mm. long; petals white, about 4 mm. long; styles in fruit moderately divergent. Seems to hybridize with *S. punctata nelsoniana*.

Alaska Penin.—Alaska Range—Alta.—northwestern Mont.—Wash. Fig. 611.

14. *S. punctata* L.

Brook Saxifrage

Leaves ascending, the blades suborbicular to reniform, 2–6 cm. wide, coarsely several-toothed with crenate or dentate gland-tipped teeth, cordate at the base; scapes 1–5 dm. tall; sepals 1.25–2 mm. long; petals 3–4.5 mm. long, white; fruit purple, 5–8 mm. long. Common and variable, represented in our area by 3 local races.

Ssp. *nelsoniana* (D. Don) Hult. (*S. nelsoniana* D. Don) is characterized by the leaves being pubescent on both surfaces. It is found in the Bering Sea and Arctic regions eastward. Fig. 612.

Ssp. *pacifica* Hult. (*S. aestivalis* auct.) of the Pacific Coast from Unalaska eastward has glabrous leaves sometimes ciliate on the margins and decidedly clavate filaments.

Ssp. *insularis* Hult. has unusually thick glabrous leaves, linear or only slightly clavate filaments, petals usually purplish, the pedicels viscid-pubescent, and occurs in the Alaska Penin., Aleutian and Shumagin Isls.

Entire species is circumboreal.

15. *S. unalaschensis* Sternb.

Unalaska Saxifrage

*Micranthes flabellifolia* (R. Br.) Small.

Leaves ascending, the blades flabellate, ciliate on the margins, glabrous or somewhat pubescent on the surfaces, the apex with a few teeth that are usually directed forward, the base narrowed and petiole-like; scapes 5–16 cm. tall, sometimes curved, purple, glandular-villous; flowers 1–9; sepals purple, about 2.5 mm. long; petals white or purplish, about 4 mm. long; carpels 2–5, in fruit erect and 7–10 mm. long.

Eastern Asia—Arctic coast—Alaska Penin.—Aleutian Isls. Fig. 613.

16. *S. integrifolia* Hook.

Hooker Saxifrage

Rootstock fibrous-rooted, stoutish; leaves 1–6 cm. long, ovate-elliptic to oblong-elliptic, entire or rarely sinuate-crenate, viscid-hirsutulous, especially on the upper surface, contracted into short, winged petioles below; scapes rather rigid, scabrous, 2–4 dm. tall; inflorescence rather narrow; sepals 1.5 mm. long; petals white about 2.5 mm. long; filaments subulate; fruit depressed, broad.

Reported from Buckland R., Vancouver Isl.—Calif.

17. *S. foliolosa* R. Br.

Foliose Saxifrage

*S. comosa* (Poir.) Britt.

Leaves crowded on the short caudex, the blades cuneate to oblanceolate with 3–5 teeth at the apex, more or less ciliate, 8–25 mm. long; scapes often more than 1, simple or branched, 6–22 cm. tall; flowers solitary at the end of the scape and often at the end of the branches, the rest of the inflorescence developing bulblets or rosules of small leaves; sepals about 1.5 mm. long; petals white, 4–5 mm. long; carpels in fruit thick, 4–5 mm. long.

Alpine-arctic, circumpolar. Fig. 614.

18. *S. ferruginea* Grah.

Alaska Saxifrage

*S. bongardii* Presl.

*S. brunoniana* Wall.

Leaves spatulate or oblanceolate, thick, hirsute on the upper surface and on the margins, sharply toothed above the middle, tapering below into a ciliate petiole, 2–10 cm. long; scapes 1–4 dm. tall, the inflorescence spreading; sepals oblong-ovate, obtuse, 1.5–2 mm. long; petals about 5 mm. long, the 3 upper differing from the lower; filaments dilated at the base. Var. *macounii* Engl. & Irmscher has many of the flowers replaced by bulblets or rosules.

Aleutian Isls.—Alta.—Mont.—Ore. Fig. 615.

19. *S. aleutica* Hult.

Aleutian Saxifrage

A peculiar, densely caespitose plant about 2 cm. tall; leaves densely congested at the end of the branches, fleshy, glabrous, ligulate, entire, 2–5 mm. long; flowers about 7 mm. in diameter; sepals and petals about equal, 2.5 mm. long; filaments filiform; fruit purplish, thick.

Known only from the high peaks of the Aleutians. Fig. 616.

20. *S. serpyllifolia* Pursh.

Thyme-leaved Saxifrage

Tufted; leaves crowded at the base of the stem, linear-spatulate, thickish, obtuse, entire, glabrous, 4–8 mm. long; stems 1-flowered, glandular, 2–6 cm. tall, with 1–3 reduced leaves; sepals ovate, about 2 mm. long; petals bright yellow, 4–7 mm. long; filaments subulate; fruit 5–7 mm. long. Var. *purpurea* Hult. has purplish petals.

Northern Asia—C. Lisburne—Alaska Range—southern Yukon—southeastern Alaska—Aleutians. Fig. 617.

21. *S. hirculus* L. Yellow Marsh Saxifrage  
*Leptasea alaskana* Small.

Basal leaves numerous, linear-oblong or linear-ovate, glabrate, entire, 1–4 cm. long; stems leafy, more or less pubescent with brown hairs, 8–25 cm. tall, mostly 1-flowered; sepals ciliate, 3–5 mm. long; petals yellow, 8–14 mm. long; carpels in fruit 8–15 mm. long.

Circumpolar with interruptions in distribution. Fig. 618.

22. *S. flagellaris* Willd. Flagellate Saxifrage

Basal leaves densely crowded, cuneate-spatulate, margined and tipped with spines, 6–16 mm. long, with many filiform runners from their axils; stems 4–15 cm. tall, glandular-pubescent, several-leaved, 1- to 5-flowered; sepals obtuse, glandular, ciliate, 3.5–5 mm. long; petals bright yellow, 7–11 mm. long.

Alpine-arctic, circumpolar. Fig. 619.

23. *S. tolmiei* T. & G. Tolmie Saxifrage

Stems leafy, trailing, glabrous, 3–10 cm. long; leaves evergreen, obovate, firm, often grooved above, the margins revolute, 4–9 mm. long; scapes 3–9 cm. tall, 1- to 4-flowered, glandular-pubescent; sepals obtuse, 2–2.5 mm. long; petals white, sometimes pinkish, 3–4 mm. long; fruiting carpels 7–10 mm. long.

Wet alpine, central Alaska—Calif. Fig. 620.

24. *S. bronchialis* L. ssp. *funstonii* (Small) Hult. Spotted Saxifrage  
*Leptasea funstonii* Small.

Tufted; leaves of the caudices crowded, persistent for several years, more or less parchment-like, linear or oblong-lanceolate with spines along the edges and tip, 6–12 mm. long; scapes with a few reduced leaves, 5–15 cm. tall, several- to many-flowered; sepals about 2 mm. long, glabrous or ciliate; petals cream-colored or yellow, spotted, 5–7 mm. long; fruiting carpels 8–10 mm. long. Var. *cherlerioides* (D. Don) Engl. of eastern Asia and the Aleutians is a form with short, very congested leaves, stems 1–4 cm. tall, petals whitish, 3 mm. long, fruit 3–4 mm. long.

Eastern Asia—Yukon, type form Eurasian. Fig. 621.

25. *S. tricuspidata* Retz. Three-toothed Saxifrage

Tufted; leaves of the caudices densely crowded, persistent, parchment-like, oblong or spatulate, with 3 sharp teeth at the apex and short-ciliate on the margins, 7–20 mm. long; scapes bearing several reduced leaves and several-many flowers; petals white or cream-color, about 6 mm. long; fruit 5–7 mm. long.

Most of Alaska—Ellesmereland—Greenl.—n. shore of L. Superior. Fig. 622.

26. *S. eschscholtzii* Sternb. Ciliate Saxifrage

Densely matted; leaves crowded, persistent, parchment-like, obovate with ciliate margins, concave above, convex below, about 1 mm. wide

and 1.5–2 mm. long; sepals ciliate, about 1 mm. long; petals none; filaments subulate; fruit 2–3 mm. long on peduncles 5–15 mm. long.

Rocky alpine, northeastern Asia—Arctic coast—central Alaska—Alaska Penin. Fig. 623.

27. *S. nudicaulis* D. Don.

Naked-stemmed Saxifrage

Leaf blades 10–25 mm. wide, reniform, cuneate to cordate at the base, 3- to 9-lobed, the lobes triangular to ovate, acute or apiculate; stipules 4–7 mm. long, ciliate; scapes 6–18 cm. tall, few- to several-flowered, the branches subtended by bracts; sepals triangular to lanceolate, 1.5–3 mm. long; petals white, 4–5 mm. long; fruit about 5 mm. long.

Bering Sea region and eastern Asia. Fig. 624.

28. *S. mertensiana* Bong.

Wood Saxifrage

Leaves 3–10 cm. wide, suborbicular with deeply cordate base, glabrate, shallowly lobed, the lobes usually with 3 rounded, gland-tipped teeth; scapes 2–4 dm. tall, glandular-pubescent, especially above, paniculately branched, the many flowers, except the terminal ones, usually replaced by bulblets; sepals 2–3 mm. long, reflexed; petals white, 3–4 mm. long; filaments clavate.

Central Alaska—western Mont.—northern Calif. Fig. 625.

29. *S. oppositifolia* L.

Purple Mountain Saxifrage

*Antiphylla oppositifolia* (L.) Small.

Tufted, densely leafy, prostrate; leaves 4-ranked, imbricated, keeled, ciliate, obovate to spatulate, 3–5 mm. long; flowers solitary on leafy stalks up to 3 cm. long; sepals ovate, ciliate, 2.5–3 mm. long; petals purplish, rarely whitish, about 8 mm. long; fruiting carpels 8–10 mm. long.

Rocky slopes, circumpolar. Fig. 626.

20. GROSSULARIACEAE (Gooseberry Family)

Shrubs; leaves palmately veined, usually lobed, petioled; flowers racemose or solitary, regular, perfect; sepals and the small petals each 5, rarely 4; stamens 5, alternate with the petals; carpels 2, united into a 1-celled ovary with 2 parietal placentae; styles 2; fruit a berry.

RIBES L.

Characters of the family. (Arabic name for *Rheum ribes*).

- |  |                            |
|--|----------------------------|
| 1A. Racemes 1- to 3-flowered.....                            | 1. <i>R. oxycanthoides</i> |
| 2A. Racemes several- to many-flowered.                       |                            |
| 1B. Stems with spines or prickles.....                       | 2. <i>R. lacustre</i>      |
| 2B. Stems unarmed.   |                            |
| 1C. Racemes 12–30 cm. long.....                              | 3. <i>R. bracteosum</i>    |
| 2C. Racemes less than 10 cm. long.                           |                            |
| 1D. Ovary and fruit smooth, fruit red.....                   | 8. <i>R. triste</i>        |
| 2D. Ovary and fruit glandular.                               |                            |
| 1E. Lower surface of leaves with resinous glands.            |                            |
| 1F. Fruit glabrous.....                                      | 4. <i>R. hudsonianum</i>   |
| 2F. Fruit puberulent.....                                    | 5. <i>R. howellii</i>      |
| 2E. Leaves not glandular, fruit prickly with stalked glands. |                            |
| 1F. Fruit black with bloom.....                              | 6. <i>R. laxiflorum</i>    |
| 2F. Fruit red.....   | 7. <i>R. glandulosum</i>   |

1. *R. oxycanthoides* L. Northern Gooseberry  
*Grossularia oxycanthoides* (L.) Mill.

Stems usually less than 1 m. tall, usually bristly, with nodal spines hardly 1 cm. long; leaves 2–4 cm. wide, cordate to widely cuneate at the base, more or less pubescent; peduncles and pedicels short, pubescent; sepals white, glabrous, 2.5–4 mm. long; petals two-thirds as long as the sepals; berry reddish-purple when ripe, about 1 cm. in diameter and of good quality.

Yukon—Newf.—Mich.—B. C. Fig. 627.

2. *R. lacustre* (Pers.) Poir. Swamp Gooseberry  
*R. echinatum* Lindl.

Stems 1–2 m. tall, more or less prickly and spiny; leaves pentagonal in outline, 5- to 7-lobed, incised-dentate, 2–7 cm. wide; petioles bristly-ciliate; flowers light green or purplish; berries black, glandular-hispid. This species is intermediate between gooseberries and currants. The fruit is used to limited extent.

Alaska Penin.—central Alaska—Labr.—Newf.—Penn.—north Calif. Fig. 628.

3. *R. bracteosum* Dougl. Blue Currant

Stems 1–3 m. tall with thick twigs; leaves cordate-orbicular in outline, 5- to 7-lobed, the lobes acute or acuminate, irregularly serrate with gland-tipped teeth, resinous-dotted beneath, 6–20 cm. long and wide; racemes with foliaceous lower bracts; flowers greenish-white; berries resinous-dotted, black with whitish bloom, 7–10 mm. in diameter, the aroma similar in an intensified degree to that of the black currant formerly grown in gardens.

South central Alaska—north Calif. Fig. 629.

4. *R. hudsonianum* Rich. Northern Black Currant

Stems 5–15 dm. tall with light gray twigs; leaves reniform-cordate, broader than long, 3- to 4-lobed, coarsely dentate, resinous-dotted and villous beneath; racemes 3–6 cm. long; bracts setaceous, villous, about equaling the pedicels, deciduous; flowers whitish; berry black, 5–10 mm. in diameter, scarcely edible.

West Alaska—Hudson Bay—Minn.—B. C. Fig. 630.

5. *R. howellii* Greene. Maple-leaved Currant  
*R. acerifolium* Howell not C. Koch.

Resembling the preceding species in general appearance but the leaves thinner and more maple-like; racemes reflexed with upturned, puberulent pedicels; sepals 3–4 cm. long, obtuse; anthers much larger than in *R. hudsonianum*. The plant from Hyder differs from the type in long pedicels and sessile or nearly sessile glands on the ovary. It may be distinct.

Hyder—Oregon.

6. *R. laxiflorum* Pursh.

Trailing Black Currant

Stems more or less decumbent, 5–20 dm. long; leaves nearly orbicular in outline, cordate, rather deeply 5-lobed, glabrous above, puberulent on the veins beneath, 5–10 cm. wide, the lobes acute, doubly serrate; racemes erect or ascending, 6- to 12-flowered, 6–10 cm. long, pubescent and glandular. The berry has a fetide odor but is often used.

Kenai Penin.—central Alaska—northern Calif. Fig. 631.

7. *R. glandulosum* Grauer.

Fetid Currant

*R. prostratum* L'Her.

Similar to *R. laxiflorum* in habit and leaf characters; odor very fetid; racemes ascending, 7- to 10-flowered, puberulent; pedicels and hypanthium glandular-bristly; berries red, 6–8 mm. in diameter.

Central Alaska—Labr.—Newf.—N. Car.—Wisc.

8. *R. triste* Pall.

American Red Currant

Stems 5–15 dm. tall with reddish-brown, shreddy bark on the twigs; leaves reniform-cordate, 3- to 5-lobed, dentate, glabrous above, glabrate or pubescent beneath, 3–10 cm. wide; flowers purplish; racemes 3–6 cm. long; fruit similar in every way to that of the cultivated garden currant.

Northern Asia—Kobuk River—Labr.—Newf.—Mich.—Ore. Fig. 632.

## 21. ROSACEAE (Rose Family)

Herbs, shrubs or trees; leaves alternate, usually with stipules; flowers regular, usually perfect but sometimes monoecious or dioecious; hypanthium well developed, ranging from flat with ovaries superior to elongated and enclosing the ovaries; sepals and petals each usually 5, the latter sometimes wanting; stamens 1–many, often 20; carpels 1–many, usually distinct; ovules 1–several in each carpel; fruit various.

## 1A. Ovary superior.

1B. Carpels 1, becoming a drupe..... 1. *Prunus*

2B. Carpels 3–5, becoming dehiscent follicles.

1C. Carpels more or less united below, shrub..... 2. *Physocarpus*

2C. Carpels distinct.

1D. Flowers dioecious, tall herb..... 5. *Aruncus*

2D. Flowers perfect.

1E. Leaves simple, shrubs..... 3. *Spiraea*

2E. Leaves twice or thrice 3-cleft..... 4. *Luetkea*

3B. Carpels becoming drupelets..... 6. *Rubus*

4B. Carpels becoming achenes.

1C. Carpels enclosed in the hypanthium which becomes fleshy in fruit..... 7. *Rosa*

2C. Carpels not enclosed.

1D. Achenes borne on a receptacle which becomes fleshy in fruit..... 8. *Fragaria*

2D. Achenes borne on a dry receptacle.

1E. Style articulate with the ovary and deciduous.

1F. Stamens numerous..... 9. *Potentilla*

2F. Stamens 5.

1G. Leaves trifoliate..... 10. *Sibbaldia*

2G. Leaves 2- to 3-ternate..... 11. *Chamaerhodos*

2E. Styles persistent.

1F. Flowers borne in a dense spicate or capitate inflorescence..... 14. *Sanguisorba*

- 2F. Flowers borne singly or in an open inflorescence.  
 1G. Leaves simple.....12. *Dryas*  
 2G. Leaves pinnate.....13. *Geum*.
- 2A. Ovary inferior.  
 1B. Leaves pinnate.....15. *Sorbus*  
 2B. Leaves simple.  
 1C. Ripe carpels bony.....18. *Crataegus*  
 2C. Ripe carpels papery or leathery.  
 1D. Cavities of the ovary as many as the pistils.....16. *Malus*  
 2D. Cavities of the ovary twice as many as the pistils. 17. *Amelanchier*

### 1. PRUNUS

Shrubs or trees; leaves simple, alternate, toothed; flowers perfect, in our species borne in racemes on leafy branches; sepals 5, imbricate; petals 5, imbricate; stamens 15-30, the filaments filiform and distinct; fruit with a fleshy exocarp and smooth bony stone. (Latin name.)

*P. melanocarpa* (A. Nels.) Shafer. Rocky Mountain Wild Cherry

Shrub or small tree; leaves glabrous, obovate or oval, usually abruptly acuminate at the apex and rounded at the base, paler beneath; flowers white, 1 cm. or less broad; fruit purple or black, 6-8 mm. in diameter, sweet or slightly astringent.

Liard Hot Springs—N. Dak.—N. Mex.—Calif.

### 2. PHYSOCARPUS Maxim.

Shrubs with exfoliating bark; leaves palmately lobed; flowers in terminal corymbs; hypanthium campanulate, 5-lobed, stellate-pubescent; sepals persistent; petals white, spreading; stamens 20-40; follicles opening along both sutures; seed 2-4, obliquely pear-shaped, shining. (Greek, bellows or bladder and fruit.)

*P. capitatus* (Pursh) Kuntze. Pacific Ninebark

1-5 m. tall; leaves 3- to 5-lobed, the lobes incised or doubly serrate, sparingly pubescent or glabrate above, sometimes stellate-pubescent beneath, 3-7 cm. long and about as wide; inflorescence rather dense; petals 3-4 mm. long; carpels 8-10 mm. long, ovate, rather long-acuminate.

Southeast Alaska—Idaho—central Calif.

### 3. SPIRÆA

Leaves without stipules; flowers small, in racemes, corymbs, or panicles; hypanthium campanulate or turbinate; sepals 5; petals 5; stamens many; carpels usually 5, inserted at the bottom of the hypanthium; ovules 2-several; fruit composed of leathery follicles which open along the ventral suture; seeds linear, tapering to both ends. (Greek, to twist, referring to the follicles of some species.)

Inflorescence conic or spike-like, petals pink..... 1. *S. menziesii*  
 Inflorescence flat to hemispherical, flowers white..... 2. *S. beauverdiana*

1. *S. menziesii* Hook.

Menzies Spiraea

An erect, branched shrub 10-15 dm. tall with reddish-brown twigs; leaves elliptic to oval, the wider forms being on the more vigorous growth,

serrate on the upper half, acute to rounded at either end, glabrous, or pubescent on the veins, 3–8 cm. long; inflorescence very dense, spike-like, 4–15 cm. long, pubescent; sepals ovate, reflexed; petals rose pink, 1.5 mm. long; follicles glabrous.

Southeast Alaska—Idaho—Ore. Fig. 633.

2. *S. beauverdiana* Schneid.

Beauverd Spiraea

*S. stevenii* (Schneid.) Rydb.

3–12 dm. tall, with reddish twigs; leaves oblong to ovate, glabrate, serrate from near the base, usually rounded at both ends, 2–5 cm. long; inflorescence 2–4 cm. across, puberulent; sepals ovate, acute, reflexed; petals white, about 1.5 mm. long; follicles puberulent.

East Asia—all of Alaska—Mackenzie. Fig. 634.

4. LUETKEA

Decumbent or creeping undershrub with stoloniferous branches; leaves twice or thrice ternately dissected; flowers borne in a raceme; hypanthium hemispheric; sepals and petals each 5; stamens about 20, the filaments subulate and connate at the base; carpels usually 5; ovules several; follicles coriaceous, dehiscent by both sutures; seed linear-lanceolate, acute. (Count F. P. Luetke was commander of a Russian exploring expedition.)

*L. pectinata* (Pursh) Kuntze.

Luetkea

Flowering shoots glabrate below, pubescent above, 5–15 cm. high; leaves crowded at the base of the flowering shoots, alternate above, glabrate, dissected into linear, acute divisions, 1–2 cm. long; racemes 1–5 cm. long; sepals ovate-lanceolate, acute, about 2 mm. long; petals white, 3 mm. or more long; carpels about 4 mm. long.

Alpine meadows, Bering Str.—Canadian Rockies—Ore. Fig. 635.

5. ARUNCUS (L.) Adans.

Perennials with thick rootstocks and twice to thrice ternate-pinnate leaves without stipules; inflorescence a large panicle, the divisions spicate; flowers dioecious; sepals 5, triangular; petals 5, narrow, white; carpels 3–5; ovules several; follicles cartilaginous, dehiscent along the ventral suture, then splitting at the apex, reflexed; seeds few. (Greek, meaning goat's beard.)

*A. vulgaris* Raf.

Goat's Beard

*A. sylvester* Kost.

*A. acuminatus* (Dougl.) Rydb.

Stem stout, glabrous, 1–2 m. tall; leaves large; leaflets lanceolate, irregularly and doubly serrate, long-acuminate, 3–12 cm. long; panicles terminal and axillary, 1–5 dm. long; flowers small; follicles about 3 mm. long.

Widely distributed in Europe, Asia, and North America. Fig. 636.



## 6. RUBUS (Tourn.) L.

Perennial herbs, shrubs or trailing vines, often prickly; leaves alternate, simple or pinnate; inflorescence axillary or terminal, the flowers solitary, racemose or paniced, regular, perfect or dioecious; stipules adnate to the petioles; sepals 5, persistent, petals 5, deciduous; stamens many, distinct; carpels few to many, inserted on a convex or elongated receptacle; fruit composed of few to many fleshy drupelets. (Latin, ruber, red.)

## 1A. Herbaceous plants.

## 1B. Flowers white.

- |                              |                          |
|------------------------------|--------------------------|
| 1C. Leaves simply lobed..... | 1. <i>R. chamaemorus</i> |
| 2C. Leaves 3-foliate.....    | 2. <i>R. pubescens</i>   |
| 3C. Leaves 5-foliate.....    | 3. <i>R. pedatus</i>     |

## 2B. Flowers pink or red.

- |  |                         |
|--|-------------------------|
| 1C. Leaves 3-lobed.....                        | 4. <i>R. stellatus</i>  |
| 2C. Leaves 3-foliate.....                      |                         |
| 1D. Stem smooth, leaflets small.....           | 5. <i>R. arcticus</i>   |
| 2D. Stem glandular-hairy, leaflets larger..... | 6. <i>R. alaskensis</i> |

## 2A. Stems woody.

## 1B. Stems biennial.

- |                        |                          |
|------------------------|--------------------------|
| 1C. Stems bristly..... | 7. <i>R. strigosus</i>   |
| 2C. Stems prickly..... | 8. <i>R. leucodermis</i> |

## 2B. Stems perennial.

- |                          |                           |
|--------------------------|---------------------------|
| 1C. Leaves simple.....   | 9. <i>R. parviflorus</i>  |
| 2C. Leaves compound..... | 10. <i>R. spectabilis</i> |

1. *R. chamaemorus* L.

## Cloudberry. Baked-apple Berry

Erect from a creeping rootstock, 5–20 cm. tall; leaves 2 or 3, reniform with 3 or 5 rounded lobes, rugose, 3–10 cm. wide; stipules ovate, obtuse; flowers solitary, dioecious; sepals ovate, glandular pubescent; petals white, obovate, 8–12 mm. long; fruit composed of 6–18 rather large drupelets the color of a baked apple when ripe and prized by the Indians and Eskimo.

Circumpolar, south to Newf.—N. Hamp.—Vancouver Isl. Fig. 637.

2. *R. pubescens* Raf.

## Dwarf Red Blackberry

Stems slender and with trailing shoots 1–10 dm. long; leaves ternate, rarely quinate; leaflets 2–9 cm. long, the lateral obliquely ovate, the terminal rhomboid, sharply and doubly serrate; flowers 1–3; petals small, white or pink; sepals pubescent, reflexed; droplets few, large, red.

Watson Lake—Newf.—New Jersey—Colo.—B. C.

3. *R. pedatus* Smith.

## Five-leaved Bramble

A slender trailing vine rooting at the nodes, glabrate; flowering branches very short, 2- to 4-leaved; leaves 3-foliate but the lateral leaflets so deeply cleft as to appear 5-foliate; leaflets thin, obovate or rhombic, irregularly toothed and incised, 1–3 cm. long; stipules ovate, small; flowers usually solitary; sepals foliaceous, ovate-lanceolate; petals white, ovate-oblong, 1 cm. or less long; fruit composed of 1–6 red, oblong drupelets.

Woods, climbing over moss or logs, eastern Asia—Yukon—Mont.—Ore. Fig. 638.

4. *R. stellatus* Smith.

Nagoon Berry

Plant low, 5–15 cm. tall from a spreading rootstock, simple or branched from the base; leaves reniform in outline, 3-lobed, sometimes divided to near the base, simple or doubly serrate, cordate at the base; stipules obovate, acuminate, strongly veined; flowers solitary; sepals lanceolate, acute, pubescent and often toothed; petals rose-red, clawed, 15–20 mm. long; fruit of high quality, composed of about 15–25 red drupelets to which the calyx strongly adheres.

Wet places in coastal districts and occasionally in interior, East Asia—B. C. Fig. 639.

5. *R. articus* L.

Nagoon Berry. Kneshenaka

*R. acaulis* Michx.

Like *R. stellatus* in habit and fruit; less than 1 dm. tall in exposed places, or to 25 cm. tall in sheltered situations; leaves 3-foliate; terminal leaflet ovate to rhombic, unevenly serrate, 2–4 cm. long; lateral leaflets oblique; flowers 1–3; petals dark rose to red; drupelets 20–40, red. *R. acaulis* was the name applied to a dwarf form with more rounded leaflets and the hypanthium part of the calyx glabrous or nearly so, the corresponding part in typical *R. articus* being glandular-hairy. Intermediate forms occur.

Circumpolar, south to north Minnesota. Fig. 640.

6. *R. alaskensis* Bailey.

Alaska Bramble

Stems 2–5 dm. tall, often woody at the base, 1- to 3-flowered, pubescent; leaves mostly 3-foliate, the petioles pubescent; terminal leaflet broadly ovate, serrate-dentate, thinly pubescent beneath, 4–8 cm. long; lateral leaflets similar but oblique; sepals narrow, 10–15 mm. long, becoming reflexed; petals pink, broadly spatulate, 12–18 mm. long.

Curry—Matanuska—southeastern Alaska. Fig. 641.

7. *R. strigosus* Michx.

American Red Raspberry

*R. idaeus* L. var. *canadensis* Rich.*R. idaeus* L. ssp. *sachalinensis* (Levl.) Focke.*R. subarcticus* Rydb.

Canes 6–12 dm. tall, brownish red, densely covered with both rough and fine bristles; leaflets 3–5, irregularly and doubly serrate, whitish-pubescent beneath; stipules very narrow and deciduous; petioles and peduncles more or less glandular; sepals triangular-lanceolate, glandular-pubescent; petals white, about 5 mm. long; fruit composed of red drupelets, elongate-hemispheric.

Across N. America, south to Conn.—Colo.—B. C., ?eastern Asia. Fig. 642.

8. *R. leucodermis* Dougl.

Western Black Raspberry

Stems 1–2 m. tall, glaucous, armed with stout, flat prickles; leaflets 3–5, ovate to lanceolate, doubly serrate, white-tomentose beneath, the veins and petioles prickly; sepals lanceolate, long-acuminate, in fruit

spreading or reflexed; petals white, shorter than the sepals; fruit usually dark with white bloom and agreeable flavor.

Southeast Alaska—Mont.—Utah—Calif.

9. *R. parviflorus* Nutt.

Thimbleberry

*R. nutkanus* Moc.

An unarmed shrub with shreddy bark, 6–16 dm. tall; leaves pentagonal in outline, 3- to 7- but mostly 5-lobed, coarsely and unevenly serrate with gland-tipped teeth, 7–20 cm. wide; sepals broadly ovate, abruptly narrowed into a long, slender appendage; petals white, 16–25 mm. long; fruit convex, red, composed of numerous small drupelets.

Southeastern Alaska—S. Dak.—N. Mex. Fig. 643.

10. *R. spectabilis* Pursh.

Salmonberry

Usually more or less prickly, 1–4 m. tall, the bark yellowish-brown and exfoliating; leaflets 3, usually more or less lobed, the lateral ones unsymmetrical, coarsely and unevenly serrate, 2–12 cm. long; stipules linear or subulate, pubescent; flowers solitary; sepals deltoid-lanceolate, pubescent; petals red, 16–22 mm. long; fruit varying from yellow to dark red, 16–25 mm. in diameter, composed of 20–40 drupelets.

East Asia—Idaho—Calif. Fig. 644.

7. ROSA (Tourn.) L.

Erect or climbing shrubs; leaves alternate, pinnate; leaflets serrate; stipules adnate; flowers perfect, pink in our species; hypanthium well developed, elongated upward, contracted at the mouth and enclosing the achenes, becoming fleshy in fruit; sepals usually 5; petals normally 5 but may be numerous by transformation of stamens; stamens numerous, inserted on the margin of the hypanthium; carpels numerous, borne on the base and sides of the hypanthium; achenes bony. (The Latin name.)

1A. Fruit 1 cm. or less in diameter..... 3. *R. woodsii*

2A. Fruit more than 1 cm. in diameter.

1B. Stems with numerous terete prickles..... 1. *R. acicularis*

2B. Stems with few flattened prickles..... 2. *R. nutkana*

1. *R. acicularis* Lindl.

Prickly Rose

Bushy, 3–12 dm. tall, usually armed with moderately strong spines interspersed with weaker ones; stipules pubescent and with glandular margins; leaflets 3–9, usually 5, elliptic or oval, regularly serrate, 15–55 mm. long, glabrous above, pale and pubescent beneath; hypanthium glabrous, pyriform, or elliptic to nearly globose, usually with a neck; sepals pubescent, glandular along the margins of the usually more or less foliose tips; petals obcordate, rose pink, 2–3 cm. long; fruit edible. Sometimes hybridizes with the next species.

Has an interrupted circumboreal distribution south to Mass.—Penn.—Colo. Fig. 645.

2. *R. nutkana* Presl.

Nootka Rose

*R. aleutensis* Crepin.

Stems stout, erect, 6–25 dm. tall, usually armed with paired straight or slightly curved prickles; stipules and leaf-rachis glandular, the stipules with glandular-dentate margins; leaflets 5–9, more or less double-serrate, usually rounded at both ends, 15–50 mm. long; sepals 15–30 mm. long, petals typically rose pink, 20–35 mm. long; fruit glabrous, typically globose and neckless.

Coastal districts, Aleutians—Calif. Fig. 646.

3. *R. woodsii* Lindl.

Woods Rose

Bushy, 5–15 dm. tall, armed with numerous prickles 4–8 mm. long; stipules narrow below the spreading tips, leaflets 5–9, obovate, somewhat cuneate at the base, slightly petioluled, serrate, glabrous, the under surface glaucous, 1–2 cm. long; flowers solitary or 2 or 3 together; sepals lanceolate, caudate-attenuate, about 15 mm. long, usually glabrous on the back, tomentose on the margin and within; fruit globose or nearly so.

Circle Hot Springs—Alta.—Minn.—Kans.—Utah. Fig. 647.

8. FRAGARIA L.

Acaulescent perennials with thick, scaly rootstocks propagating by runners which root at the joints; bractlets, sepals, and petals each usually 5; flowers usually white; stamens about 20; receptacle hemispheric or conic, bearing the numerous carpels and becoming enlarged and fleshy in fruit; styles filiform but short and attached near the middle of the ovaries. (Latin, signifying fragrance.)

1A. Leaves thick and coriaceous..... 1. *F. chiloensis*

2A. Leaves thinner.

1B. Pubescence of stems and petioles spreading or slightly

reflexed ..... 2. *F. bracteata*

2B. Pubescence ascending or appressed..... 3. *F. glauca*

1. *F. chiloensis* (L.) Duch.

Beach Strawberry

Rather stout; petioles, peduncles and inflorescence silky-pubescent with spreading or reflexed hairs; leaflets thick, cuneate-obovate or the lateral rhombic, crenate-dentate, rugose above, silky-pubescent beneath, 2–4 cm. long; peduncles shorter than the leaves; sepals acuminate; flowers 2–3 cm. broad; fruit ovoid, up to 25 mm. long, soft and sweet; achenes nearly superficial.

Near the coast, Aleutians—Calif.—Peru—Patagonia, and in Hawaii. Fig. 648.

2. *F. bracteata* Heller.

Bracted Strawberry

Rootstock short; leaves thin, silky when young, nearly glabrous in age; leaflets broadly ovate, coarsely serrate, 2–4 cm. long; scapes slender, equaling or exceeding the leaves, usually with a unifoliate bract; flowers 15–20 mm. broad; sepals triangular-lanceolate, longer than the lanceolate bractlets, very acute; fruit ovoid, the achenes nearly superficial.

Hyder—Mont.—N. Mex.—Calif. Fig. 649.

3. *F. glauca* (Wats.) Rydb.

Yukon Strawberry

*F. yukonensis* Rydb.*F. platypetala* of reports from Alaska.

Rather slender; petioles and peduncles appressed-villous; leaflets rather thin, obovate, cuneate at the base, sharply and deeply toothed, glabrous above, appressed silky beneath, 15–55 mm. long; scapes leafy-bracted, usually shorter than the leaves; flowers less than 15 mm. wide; fruit subglobose, about 1 cm. in diameter; achenes in shallow pits.

Central Alaska—Gt. Slave L.—Black Hills—N. Mex. Fig. 650.

## 9. POTENTILLA L.

Herbs or rarely shrubs with alternate, compound leaves; flowers regular, perfect; hypanthium concave to hemispheric; bractlets, sepals and petals each 5; stamens usually many; receptacle hemispheric or conic, bearing many carpels; styles terminal or lateral; fruit composed of many achenes on a dry receptacle. (Latin, powerful, from medicinal properties of some species.)

- 1A. Petals purple, short. (*Comarum* L.) ..... 1. *P. palustris*
- 2A. Petals white or cream color. (*Drymocallis* Fourr.) ..... 2. *P. arguta*
- 3A. Petals yellow.
  - 1B. Plant shrubby. (*Dasiphora* Raf.) ..... 3. *P. fruticosa*
  - 2B. Plant herbaceous.
    - 1C. Plant stoloniferous, flower solitary. (*Argentina* Lam.)
      - 1D. Bractlets toothed or divided, achenes grooved ..... 4. *P. anserina*
      - 2D. Bractlets entire, achenes not grooved ..... 5. *P. pacifica*
    - 2C. Plants lacking runners, flowers in cymes.
      - 1D. Leaves odd-pinnate.
        - 1E. Leaves with 3–7 pairs of leaflets.
          - 1F. Leaves silky-tomentose on both sides ..... 6. *P. hippiana*
          - 2F. Leaves green or grayish on upper side ..... 7. *P. pennsylvanica*
        - 2E. Lower leaves with 2 or 3 pairs of leaflets.
          - 1F. Style filiform.
            - 1G. Leaflets pinnatifid ..... 11. *P. multifida*
            - 2G. Leaflets toothed ..... 12. *P. rubricaulis*
          - 2F. Style enlarged and glandular at the base.
            - 1G. Leaves silky-pubescent on both sides ..... 10. *P. pulchella*
            - 2G. Leaves tomentose beneath, green above.
              - 1H. Leaflets pinnatifid almost to the midrib .... 9. *P. virgulata*
              - 2H. Leaflets pinnatifid  $\frac{1}{2}$ – $\frac{3}{4}$  way to midrib .... 8. *P. pectinata*
        - 2D. Leaves palmately 5- to 7-foliate.
          - 1E. Leaflets toothed to the base ..... 14. *P. gracilis*
          - 2E. Leaflets toothed on upper half only ..... 13. *P. diversifolia*
    - 3D. Leaves trifoliate.
      - 1E. Leaflets cleft to the middle or lower (see also *P. vahliana*).
        - 1F. Petals 2–4 mm. long ..... 16. *P. elegans*
        - 2F. Petals 5–8 mm. long ..... 15. *P. biflora*
      - 2E. Leaflets toothed.
        - 1F. Leaves hirsute on lower surface.
          - 1G. Plant erect ..... 18. *P. monspeliensis*
          - 2G. Plant spreading ..... 17. *P. emarginata*
        - 2F. Leaves tomentose or densely sericeous on lower surface.
          - 1G. Stems 1 dm. or less tall, 1- to 3-flowered.
            - 1H. Petals obcordate ..... 20. *P. uniflora*
            - 2H. Petals obreniform ..... 21. *P. vahliana*
          - 2G. Stems normally 1–2 dm. tall, several-flowered.
            - 1H. Flowers 2–3 cm. in diameter ..... 19. *P. villosa*
            - 2H. Flowers 15 mm. or less in diameter.
              - 1J. Leaves deeply dissected ..... 22. *P. hookeriana*

- 2J. Leaves coarsely dentate..... 23. *P. nivea*  
 2G. Stems more than 25 cm. tall..... 24. *P. chamissonis*

1. *P. palustris* (L.) Scop. Purple or Marsh Cinquefoil  
*Comarum palustre* L.

Aquatic or marsh perennial with creeping rootstocks; stems ascending, more or less hirsute and glandular-pubescent above; leaves pinnate, leaflets 3-7, usually 5, green above, pale beneath, oblong or oval, sharply serrate, 2-6 cm. long; bractlets small and narrow; sepals purple, ovate, acuminate, 8-15 mm. long; petals much shorter than the sepals; style lateral; achene smooth with purplish apex.

Circumboreal, south to Penn.—Wyo.—Calif. Fig. 651.

2. *P. arguta* Pursh. Tall or Glandular Cinquefoil  
*Drymocallis arguta* (Pursh) Rydb.

Rootstock stout and woody; stems stout, erect, 3-10 dm. tall, striate, hirsute, glandular or viscid; basal leaves 7- to 11-foliate; leaflets ovate, oval or rhomboid, the terminal one cuneate, the lateral ones oblique, all sharply incised-dentate; stem leaves reduced; flowers in a dense cyme, 12-18 mm. in diameter; hypanthium, bractlets and calyx glandular viscid; petals whitish, drying yellowish, a little longer than the sepals.

Yukon—N. B.—Va.—Colo. Fig. 652.

3. *P. fruticosa* L. Shrubby Cinquefoil. Yellow Rose  
*Dasiphora fruticosa* (L.) Rydb.

A much-branched shrub with shreddy bark, 2-12 dm. tall; leaves pinnate, silky pubescent, especially beneath; leaflets usually 5, oblong or linear-oblong, entire and usually with more or less revolute margins, 10-25 mm. long; petals 10-15 mm. long, much longer than the sepals; achenes dark, receptacle with long brown hairs.

Circumpolar, south to N. Jer.—Minn.—N. Mex.—Calif. Fig. 653.

4. *P. anserina* L. Common Silverweed  
*Argentina anserina* (L.) Rydb.

Leaves 1-2 dm. long; leaflets 9-31 with smaller ones interspersed, 1-4 cm. long, oblong or oblong-lanceolate, white-silky beneath, sparingly silky to green and glabrate above; peduncles 3-15 cm. long; petals 7-15 mm. long; achenes corky, grooved on the upper end. Var. *sericea* Hayne (*Argentina argentea* Rydb.) has the upper surface of the leaves silky-tomentose.

Interrupted circumboreal, south to N. Jer.—N. Mex. Fig. 654.

5. *P. pacifica* Howell. Pacific Silverweed  
*P. yukonensis* Hult.  
*P. egedii* Wormskj. var. *groenlandica* (Tratt.) Polunin.  
*Argentina occidentalis* Rydb.  
*A. subarctica* Rydb.

Resembling the preceding in appearance; leaves up to 4 dm. long including petiole; leaflets up to 6 cm. long, glabrous or nearly so above,

silky-tomentose beneath; peduncles up to 3 dm. long; petals up to 15 mm. long. The vigorous form on the Pacific Coast gives way gradually to the diminutive form of the Arctic Coast which often has leaves and peduncles only a few centimeters high.

Mostly along beaches, circumpolar, south to Calif. Fig. 655.

6. *P. hippiana* Lehm.

Wooly Cinquefoil

Stems erect, 3–6 dm. tall, silky canescent; lower leaves 5-to 11-foliolate; leaflets oblanceolate or oblong, obtuse, narrowed or cuneate at the base, 15–50 mm. long, deeply toothed; sepals ovate-lanceolate, 5–7 mm. long; bractlets nearly equaling the sepals but narrower; petals 6–8 mm. long.

Central Alaska—Minn.—Nebr.—Ariz.

7. *P. pennsylvanica* L.

Pennsylvania Cinquefoil

Stems erect or ascending, 4–8 dm. tall in the typical form, more or less tomentose; leaves pinnately 5- to 15-foliolate; leaflets oblong or oblanceolate, cleft one-half way to the midrib into oblong divisions, grayish tomentose and veiny beneath, glabrous or nearly so above; bractlets about equaling the sepals; petals longer than the sepals; achenes smooth or more often somewhat rugulose. Var. *strigosa* Pursh is generally lower, 3–5 dm. tall, leaflets deeply divided into narrow lobes with revolute margins. Var. *glabrata* Wats. has stem and leaves nearly glabrous.

Asia, east central Alaska—Hudson Bay—Kans.—N. Mex. Fig. 656.

8. *P. pectinata* Fisch.

Coast Cinquefoil

Stems usually clustered from a woody rootstock, finely pubescent, 2–5 dm. tall; stipules large, foliaceous and lobed; leaves mostly 5-foliolate; leaflets obovate or oblong, cut into narrow lobes with revolute margins, 2–5 cm. long; flowers about 15 mm. in diameter; bractlets lanceolate with narrowed bases; sepals lanceolate with broad bases, a little longer than the bractlets; achenes smooth or minutely rugulose.

Skagway and in eastern North America. Fig. 657.

9. *P. virgulata* A. Nels.

Caudex short and with a taproot; stems 2–5 dm. tall; leaflets ovate, 1–4 cm. long, sparingly hairy above, white pubescent beneath, dissected into narrowly linear divisions with revolute margins; bractlets linear, about equaling the lanceolate sepals; petals somewhat exceeding the sepals; achenes smooth.

Seward Penin.—Wyo.—Utah. Fig. 658.

10. *P. pulchella* R. Br.

Densely caespitose and silky-hirsute with white or yellowish hairs; stems spreading, 1- to few-flowered, less than 1 dm. long; leaves usually 5-foliolate; leaflets obovate-cuneate, deeply dissected into linear segments; bractlets oblong, nearly as long as the ovate sepals; petals 5–6 mm. long, a little exceeding the sepals; styles short.

Wrangel Isl.—Ellsmereland—Spitzbergen — Nova Zemlya — Labr. — Seward Penin.—?Kiska Isl.

11. *P. multifida* L.

Cut-leaved Cinquefoil

Stems, several to many, arising from a woody caudex, ascending or spreading, somewhat appressed-strigose, 1–3 dm. long; leaflets pectinately divided to very near the midrib into narrow, linear divisions with more or less revolute margins, smooth above, tomentose beneath; bractlets slightly shorter and petals slightly longer than the 3–4 mm. long sepals; style short; achenes smooth or somewhat rugose.

Circumpolar, south to Great Slave L.—southern Alaska. Fig. 659.

12. *P. rubricaulis* Lehm.

Red-stemmed Cinquefoil

Stems several, ascending or prostrate, often tinged with red, 1–2 dm. long, pubescent with spreading hairs; leaflets glabrate above, white tomentose beneath, 1–3 cm. long, obovate or oblanceolate, pinnately cleft into lanceolate, acute teeth; stem leaves usually ternate; cymes 5- to 9-flowered; petals obcordate, a little longer than the sepals.

Reported from Herschel Isl., Mackenzie—Ellsmereland—Great Bear Lake.

13. *P. diversifolia* Lehm.

Diverse-leaved Cinquefoil

*P. glaucophylla* of reports from Alaska.

Stems 1–few from a woody caudex, 2–5 dm. tall; leaflets pubescent when young, often glabrate in age, obovate or oblanceolate, toothed or lobed on the upper half, 2–5 cm. long; stipules of basal leaves lanceolate and scarious, of upper leaves wider and foliaceous; bractlets shorter than the sepals; petals obcordate, 5–9 mm. long; styles long, filiform.

Alaska Range—S. Dak.—Colo.—Calif. Fig. 660.

14. *P. gracilis* Dougl.

*P. alaskana* Rydb.

*P. blaschkeana* and *P. nuttallii* of reports from Alaska.

Stems pubescent, branched above, 4–9 dm. tall; basal leaves long-petioled; leaflets obovate, cut one-half way to the midrib into narrow lobes, pubescent, sometimes silky, especially beneath, 3–12 cm. long; inflorescence silky; sepals 8–10 mm. long, longer than the bractlets; petals about equaling the sepals; achenes smooth; base of style dilated.

Kodiak Isl.—Wiseman—Alta.—Mont.—Calif. Fig. 661.

15. *P. biflora* Willd.

Two-flowered Cinquefoil

Almost acaulescent, caespitose, silky pubescent alpine plant; stipules linear-lanceolate; terminal leaflet split nearly to the base into 3 linear divisions, the lateral into 2 such divisions, all pubescent beneath, glabrate above in age, the margins revolute; scapes 3–10 cm. tall, 1- or 2-flowered; bractlets and sepals about equal; achenes nearly 2 mm. long; receptacle with long silky pubescence.

Eastern Asia—Mackenzie River—Alaska Range. Fig. 662.



16. *P. elegans* C. & S.

Pretty Cinquefoil

Densely caespitose or pulvinate; stems 15–30 mm. tall, 1-flowered; leaves short petioled, the leaflets 3–6 mm. long, sparsely villous-pilose; petals slightly exceeding the sepals and bracts. A very small and delicate species.

Eastern half of Asia, extending to Mackenzie.

17. *P. emarginata* Pursh.

Arctic Cinquefoil

*P. nana* Willd.

Caespitose; 2–15 cm. tall; leaflets sessile, softly hirsute, 3- to 9-toothed, 5–15 mm. long; stems 1- or 2-flowered; bractlets 4–5 mm. long, about equaling the ovate, acute sepals; petals broadly obcordate, 5–9 mm. long; style filiform, short; achenes glabrous.

Circumpolar, south to Labr.—southern Alaska—Aleutians. Fig. 663.

18. *P. monspeliensis* L.

Rough Cinquefoil

*P. norvegica* ssp. *monspeliensis* (L.) Achers. & Graebn.

Stems erect from an annual or biennial root, branched, hirsute, 2–8 dm. tall; stipules foliaceous, entire or dentate; leaves trifoliate, rarely 5-foliate on young, vigorous growth; leaflets variable, usually obovate, deeply serrate, pubescent with spreading hairs, 2–6 cm. long; flowers in rather dense, leafy-bracted cymes; bractlets and sepals lanceolate, acute; petals nearly as long as the sepals; achenes rugulose.

Widespread in our area, Alaska—Labr.—Mexico—Calif. Fig. 664.

19. *P. villosa* Pall.

Villous Cinquefoil

Silky-villous throughout; stems 15–30 cm. tall, 1- to several-flowered; leaflets with prominent veins, greenish above, silvery beneath, obovate, deeply crenate-dentate with rounded teeth, 1–5 cm. long; bractlets acute; sepals acute, broader and slightly longer than the bractlets, 6–8 mm. long; petals 8–12 mm. long; achenes nearly smooth but generally with a few lines.

Eastern Asia—Seward Penin.—Alaska Range—Aleutians. Fig. 665.

20. *P. uniflora* Ledeb.

One-flowered Cinquefoil

Silky-pubescent, stems 3–12 cm. tall, usually 1-flowered but sometimes 2-flowered; leaflets silky or glabrate above, white-tomentose beneath, deeply cut from the apex, the terminal one cuneate-obovate, the lateral ones rhombic, 1–2 cm. long; bractlets and sepals silky, the bractlets obtuse, the sepals acute, 4–5 mm. long; petals obcordate, 6–8 mm. long.

Lena River, Siberia—Alta.—Mont.—Colo.—Ore.—Kamchatka. Fig. 666.

21. *P. vahliana* Lehm.

Vahl Cinquefoil

Caudex woody, covered with old remains of stipules and petioles; whole plant covered with yellowish villous hairs; leaves crowded, short petioled; leaflets usually 1 cm. or less long, cuneate, coarsely and deeply

dentate at the apex; bractlets broadly ovate or elliptic, often obtuse; petals usually broader than long and overlapping.

Wrangel Isl.—Ellsmere land—Labr.—St. Matthew Isl. Fig. 667.

22. *P. hookeriana* Lehm.

Hooker Cinquefoil

Caespitose; stems 1–2 dm. tall, tomentose; basal leaves on petioles 1–3 cm. long; leaflets 1–2 cm. long, deeply cleft into oblong lobes, silky villous above, densely tomentose beneath; bractlets almost as long as the sepals which are about 4 mm. long; petals obcordate, slightly exceeding the sepals.

Urals—Victoria Land—Mont. Fig. 668.

23. *P. nivea* L.

Snow Cinquefoil

Caespitose, the caudex covered with the brown stipules and old leaves; stems several, 10–25 cm. tall, more or less tomentose, few-leaved; basal leaves on petioles 2–5 cm. long; leaflets oblong-cuneate or obovate, 15–30 mm. long, glabrate or slightly villous above, densely white-tomentose beneath, coarsely and deeply crenate; sepals ovate-lanceolate, longer than the bractlets and shorter than the petals which are narrowly obcordate.

Circumboreal, Yukon, south to Colo.—Nevada. Fig. 669.

24. *P. chamissonis* Hult.

Chamisso Cinquefoil

Stems several, 2–5 dm. tall; lower leaves long-petioled, 3-foliate or a few 5-foliate; leaflets obovate, the lateral sessile, the terminal long petiolulate, deeply serrate-dentate; inflorescence many-flowered; bracts linear to lanceolate, shorter than the narrowly triangular sepals; achenes about 1 mm. long, the style being of about same length and papillose at the base.

Southern Yukon — Quebec — Greenland — Spitzbergen — northern Scandinavia.

# 10. SIBBALDIA L.

Low, tufted perennials with woody caudices; leaves ternate; flowers in cymes on scape-like, nearly leafless stems; bractlets, sepals, and petals each 5; petals obovate, yellow, shorter than the sepals; stamens 5, inserted alternate with the petals on the wooly edge of the hypanthium; carpels 5–20; styles lateral; achenes glabrous. (Robert Sibbald was a Scotch naturalist.)

*S. procumbens* L.

Sibbaldia

Stems pubescent, less than 1 dm. tall; leaflets more or less appressed-pubescent, obovate, cuneate at the base, 2- to 5- but usually 3-lobed at the apex, 1–3 cm. long; sepals slightly longer than the bractlets, acute or acuminate.

Alpine-arctic, circumboreal, south to Newf.—N. Hamp.—Colo.—Calif. Fig. 670.

## 11. CHAMAERHODOS Bunge.

Perennial or biennial herbs; leaves ternately divided; flowers small, perfect, borne in cymes; bractlets wanting; hypanthium cup-shaped, small; sepals and petals each 5, stamens 5, opposite the petals; pistils 5-20; style filiform, basal. (Greek, a low rose.)

*C. nuttallii* (T. & G.) Pickering.

American Chamaerhodos

*C. erecta* (L.) Bunge ssp. *nuttallii* (T. & G.) Hult.

Usually much branched, hirsute and glandular; basal leaves 2- to 4-ternately divided into linear or oblong segments; stem leaves diminishing in size and complexity upward; flowers numerous; hypanthium 2-3 mm. in diameter; sepals triangular-lanceolate, about equaling the white petals.

Yukon—L. Athabasca—Manitoba—Minn.—Colo. Fig. 671.

## 12. DRYAS L.

Low tufted or matted subshrubs; leaves alternate, petioled, simple, more or less rugose, white-tomentose beneath; flowers solitary on naked peduncles; bractlets wanting; sepals 7-10, persistent; petals 7-10, longer than the sepals, often persistent; stamens numerous; carpels numerous; style terminal, elongating and becoming plumose in fruit. (Latin name of a Greek wood nymph.)

1A. Sepals ovate or ovate-lanceolate, petals yellow

and ascending..... 1. *D. drummondii*

2A. Sepals linear or linear-lanceolate, petals whitish, spreading.

1B. Leaf-blades crenate, strongly rugose..... 2. *D. octopetala*

2B. Leaf-blades entire or with a few teeth, not  
conspicuously rugose..... 3. *D. integrifolia*

1. *D. drummondii* Rich.

Drummond Mountain Avens

Often forming mats several decimeters in diameter; leaves elliptic, narrowed at the base, the margins slightly revolute, 1-3 cm. long; peduncles 5-20 cm. tall, tomentose; sepals black glandular-pubescent, about 5 mm. long; petals obovate, about twice as long as the sepals; achenes with plumes up to 4 cm. long.

Central and southern Alaska—Great Bear L.—Mont.—Ore., and in Ontario and Quebec. Fig. 672.

2. *D. octopetala* L.

Eight-petaled Mountain Avens

Densely tufted; leaves elliptic, glabrous and rugose above, the margins revolute, rounded at the apex, rounded or subcordate at the base, 1-3 cm. long; peduncles 3-15 cm. long, tomentose, often black hairy on upper part; sepals black glandular-pubescent, about 7 mm. long; petals about 1 cm. long; achenes with plumes up to 3 cm. long. Variable and consists of several races or varieties. Hybridizes with *D. integrifolia*.

Circumpolar, south to Colo. Fig. 673.

3. *D. integrifolia* Vahl.

Entire-leaved Mountain Avens

Similar to *D. octopetala*; leaves only slightly rugose, ovate or ovate-lanceolate, the revolute margins entire or with a few teeth near the base,

the apex sometimes acute; sepals acute. Var. *sylvatica* Hult. is a shade form with narrower leaves up to 45 mm. long; peduncles in fruit up to 20 cm. long.

Eastern Asia—Ellesmereland—Greenl.—Newf.—N. Hamp.—B. C. Fig. 674.

### 13. GEUM L.

Perennials; leaves pinnate, in some species the terminal leaflet much the largest; flowers yellow or whitish; bractlets, sepals and petals each 5; stamens many, filaments capillary; carpels many on a conical or clavate receptacle; style persistent. (The ancient Latin name.)

- 1A. Style conspicuously bent and geniculate above..... 1. *G. macrophyllum*
- 2A. Style not conspicuously bent or geniculate (*Sieversia* Willd.).....
- 1B. Style not much elongated in fruit..... 2. *G. rossii*
- 2B. Style elongating in fruit and plumose below.
- 1C. Basal leaves with terminal leaflet much the largest... 3. *G. calthifolium*
- 2C. Basal leaves pinnate with leaflets of nearly same size.
- 1D. Leaflets 5-7..... 4. *G. pentapetalum*
- 2D. Leaflets 11-17..... 5. *G. glaciale*

#### 1. *G. macrophyllum* Willd. Large-leaved Avens

Stem more or less hirsute, 4-9 dm. tall; basal leaves interruptedly pinnate with a large terminal cordate, doubly crenate-dentate leaflet 5-10 cm. broad; stem leaves 3-foliate or deeply 3-lobed; all leaflets more or less hirsute on both sides; petals ovate, longer than the reflexed calyx lobes; receptacle and ovary pubescent; style curved and jointed, the lower portion of upper joint pubescent; achenes hooked. Ssp. *perincisum* (Rydb.) Hult. has narrower, more deeply incised, acute leaflets with longer, more acute teeth.

The species in the coast regions and the subspecies in interior Alaska, eastern Asia—Newf.—N. Hamp.—Colo.—Ariz.—Calif. Fig. 675.

#### 2. *G. rossii* (R. Br.) Ser. Ross Avens *Sieversia rossii* R. Br.

Stems arising from a large, upright, woody caudex, 7-25 cm. tall; basal leaves interruptedly pinnate, 5-10 cm. long including petiole; larger leaflets 9-15, variously incised and toothed, pubescent on the margins, 7-15 mm. long; stems with about 3 reduced leaves, 1- or 2-flowered; sepals and bractlets lanceolate, pubescent; petals bright yellow, about 1 cm. long and broad.

Alpine-arctic, eastern Asia—Melville Isl.—Yukon—Aleutians. Fig. 676.

#### 3. *G. calthifolium* Menz. Caltha-leaved Avens *Sieversia calthifolia* (Menz.) D. Don

Hirsute; rootstock thick, nearly horizontal; stems 1-3 dm. tall, scape-like, with a few reduced leaves; basal leaves of one large, cordate-reniform, doubly crenate, often slightly lobed leaflet, 3-10 cm. wide, and a few much reduced lateral ones; flowers 1-few; sepals lanceolate, acute, hirsute, 8-10

mm. long; petals broad, usually emarginate, 8–14 mm. long; achenes hirsute, the developed plumose style 10–15 mm. long.

Coast regions, eastern Asia—Aleutians—Vancouver Isl. Fig. 677.

*G. calthifolium* × *G. rossii* (*Sieversia macrantha* Kearney) occurs where the ranges of the two species overlap. Stems 1–4 dm. tall, more or less pubescent, branched above; basal leaves up to 14 cm. long; leaflets 7–13, the upper one deeply lobed, the lowermost reduced, all irregularly serrate; flowers and fruit intermediate between the parents.

4. *G. pentapetalum* (L.) Makino.

Low Avens

*Sieversia pentapetala* (L.) Greene

Base more or less suffruticose; leaves glabrous, crowded at the end of the branches; leaflets 5–7, cuneate or ovate-lanceolate, toothed toward the apex, 5–15 mm. long; peduncles 3–10 cm. long; bractlets shorter than the sepals; sepals ovate-lanceolate, acuminate, 6–8 mm. long; petals about 1 cm. long, very light yellow.

Japan—eastern Siberia—Aleutians. Fig. 678.

5. *G. glaciale* Adams.

Glacier Avens

*Sieversia glacialis* (Adams) Spreng.

Rootstocks short, thick, dark purplish-brown; basal leaves sparsely pilose above, densely so beneath with soft yellowish hairs; leaflets many, mostly 8–12 mm. long, often toothed, tipped with long hairs, the terminal one larger and lobed; stem leaves few and small; stems usually 1-flowered, 1–2 dm. tall, bractlets lanceolate, shorter than the sepals; sepals acute, 7–8 mm. long; petals rather light yellow, longer than the sepals.

Bering Sea and Arctic coasts, Lena R.—Mackenzie R. Fig. 679.

14. SANGUISORBA L.

Perennials with thick rootstocks; leaves odd-pinnate; flowers small, borne in dense spikes on long, naked peduncles; stipules adnate; leaflets toothed; hypanthium urn-shaped, angled, constricted at the mouth; sepals 4, petaloid; petals none; stamens 4–12 or more; carpels 1–3; style filiform, terminal; achenes usually 1, enclosed in the hypanthium. (Latin, blood and absorb.)

- |  |                          |
|--|--------------------------|
| 1A. Stamens scarcely or not at all exceeding the sepals, the filaments filiform..... | 1. <i>S. officinalis</i> |
| 2A. Stamens longer than the sepals, filaments flattened.                             |                          |
| 1B. Flowers purplish.....  | 2. <i>S. menziesii</i>   |
| 2B. Flowers greenish or whitish.....   | 3. <i>S. sitchensis</i>  |

1. *S. officinalis* L.

Official Great Burnet

*S. microcephala* Presl of some reports.

Glabrous, rather slender, 3–12 dm. tall; leaflets 7–13, oval or ovate, regularly serrate with gland-tipped teeth, on petiolules less than 1 cm. long, 1–6 cm. long; flowers dark purple in spikes 1–3 cm. long and about

1 cm. thick; sepals ovate, often minutely pubescent on the back; hypanthium and fruit 4-winged.

Bogs and wet soil, Bering Str.—Yukon, Eurasia. Fig. 680.

2. *S. menziesii* Rydb.

Menzies Great Burnet

Stems slender, 3–10 dm. tall; leaflets 9–15, rounded oval to ovate, 2–6 cm. long, coarsely serrate with broadly ovate teeth; petiolules 6–25 mm. long; spikes 1–3 cm. long; sepals dark purple, oval, about 2.5 mm. long; filaments 5–7 mm. long.

Southern Alaska—Wash. Fig. 681.

3. *S. sitchensis* C. A. Mey.

Sitka Great Burnet

*S. latifolia* (Hook.) Cov.

Leafy, 4–12 dm. tall; leaflets 7–21, ovate or elliptic, serrate with sharp-pointed teeth, cordate, 1–7 cm. long; spike dense, 2–10 cm. long, 1 cm. or more thick; flowers greenish-white, sometimes tinged with purple; sepals oval; stamens 4, long-exserted.

Wet soil, Arctic Circle—Idaho—Ore.—eastern Asia. Fig. 682.

15. SORBUS (Tourn.) L.

Trees or shrubs; ours with alternate, pinnate leaves; stipules deciduous, flowers small, perfect, regular, white, borne in terminal compound cymes; sepals 5, deciduous; styles usually 3, distinct; ovules 2 in each cell of the ovary; fruit a red berry-like pome. (The ancient Latin name for the pear or service-tree.)

- |                                    |                           |
|------------------------------------|---------------------------|
| 1A. Tree, up to 15 m. tall.....    | 4. <i>S. aucuparia</i>    |
| 2A. Shrubs, 4 m. or less tall..... |                           |
| 1B. Leaflets usually 7 or 9.....   | 1. <i>S. sambucifolia</i> |
| 2B. Leaflets usually 9 or 11.....  | 2. <i>S. sitchensis</i>   |
| 3B. Leaflets 11–15.....            | 3. <i>S. scopulina</i>    |

1. *S. sambucifolia* (C. & S.) Roem.

Elder-leaved Mountain Ash

1–2 m. tall; leaflets 7–11, 2–7 cm. long, lanceolate to ovate-lanceolate, acuminate, usually broadest at the asymmetrical base, the margins sharply serrate almost to the base; inflorescence round-topped, 8- to 15-flowered; flowers 10–15 mm. in diameter; sepals triangular, somewhat ciliolate, stamens about as long as the petals; styles 5; fruits ellipsoid, glaucescent, 10–15 mm. in diameter.

An east Asian species occurring in the western Aleutians. Fig. 683.

2. *S. sitchensis* Roem.

Sitka Mountain Ash

Usually about 1 m. tall in alpine situations but up to 4 m. at lower elevations; leaflets oval or oblong, 3–7 cm. long, the apex rounded or slightly acutish, the margins serrate on the upper one-third to two-thirds; inflorescence round-topped, 15- to many-flowered; flowers 6–9 mm. broad, fragrant; sepals ciliolate; top of ovary pubescent; fruit subglobose or ellipsoid, red, becoming orange and finally purplish, 8–10 mm. in diameter.

Pacific coast of Alaska—Mont.—B. C. Fig. 684.

3. *S. scopulina* Greene. Western Mountain Ash  
*S. alaskana* G. N. Jones not Hollick.  
*S. andersonii* G. N. Jones

1-4 m. tall; leaflets elliptic or elliptic-lanceolate, acute or acuminate, serrate from near the base, 3-8 cm. long; inflorescence many-flowered; fruit bright red, subglobose, 8-10 mm. in diameter.

Bering Sea—L. Athabasca—Black Hills—N. Mex.—Calif. Fig. 685.

4. *S. aucuparia* L. European Mountain Ash. Rowan Tree

Leaflets 9-15, oblong-lanceolate, acute, 3-5 cm. long, upper two-thirds serrate, entire toward the base; inflorescence usually 75-100-flowered; fruit scarlet, subglobose, 9-11 mm. in diameter.

A native of Europe but spreading rapidly from cultivation.

#### 16. MALUS Juss.

Trees or shrubs; leaves toothed or lobed; flowers perfect, regular, showy, white or pink; flowers in small cymes; sepals 5; petals 5, rounded and clawed; styles 2-5, united at the base; ovary 2- to 5-celled, with 2 ovules in each cell; carpels papery or leathery, enclosed in the enlarged hypanthium, forming a pome usually depressed at the base. (Greek, apple.)

- M. fusca* (Raf.) Schneider. Western Crab-apple  
*M. diversifolia* (Bong.) Roem.  
*Pyrus diversifolia* Bong.  
*Pyrus rivularis* Dougl.

A shrub or small tree, 2-5 m. tall; young growth pubescent; leaves ovate, variable, serrate, sometimes more or less lobed, glabrous above, pubescent beneath, acute, 3-8 cm. long; petals white, about 1 cm. long; calyx pubescent, not persisting in fruit; fruit usually oblong, sometimes subglobose, about 1 cm. long, acid but not astringent.

Near the coast, southern Alaska—Calif. Fig. 686.

#### 17. AMELANCHIER Medic.

Shrubs or trees; leaves simple; flowers racemose, white; sepals 5, reflexed, persistent; stamens many, inserted on the throat of the calyx; styles 3-5; ovary 3- to 5-celled becoming twice as many celled by intrusion of false partitions from the back; ovules solitary in each cell; fruit berry-like. (The Savoy name of the Medlar.)

Leaves about as broad as long..... 1. *A. alnifolia*  
 Leaves distinctly longer than broad..... 2. *A. florida*

1. *A. alnifolia* Nutt. Northwestern Service-berry

A low shrub, 1-2 m. tall; leaves thick and firm, nearly orbicular or round-oval, 2-4 cm. long, glabrous above, tomentose beneath when young; sepals densely wooly; petals oblanceolate-oblong, about 1 cm. long; fruit about 8 mm. in diameter.

Central Alaska—Sask.—Nebr.—Colo. Fig. 687.

2. *A. florida* Lindl.

Pacific Service-berry

A shrub or tree, 2–5 m. tall; leaves oblong, usually entire near the rounded base, serrulate toward the rounded apex, 2–5 cm. long; racemes 4–8 cm. long; sepals lanceolate, acute, glabrous or slightly pubescent; petals oblanceolate, 12–15 mm. long; fruit purple, juicy, 8–10 mm. in diameter.

Alaska Penin.—Oregon. Fig. 688.

## 18. CRATAEGUS L.

Shrubs or small trees, usually armed with spines; leaves simple, alternate, toothed, often lobed; flowers in corymbs, usually white; sepals 5; petals 5; stamens 5–25; carpels 1–5, separate; fruit a drupe-like pome containing 1–5 bony nutlets. (Greek, meaning strong, from the toughness of the wood.)

*C. douglasii* Lindl.

Black Hawthorn

Spines 15–25 mm. long; leaves variable, doubly serrate above the cuneate base, often slightly lobed, 2–8 cm. long, glabrous beneath, pubescent above, at least on the midrib and veins; corymbs usually many-flowered; petals orbicular, 4–5 mm. long; fruit black.

Hyder—Mich.—N. Mex.—California. Fig. 689.

## 22. FABACEAE (Pea Family)

Herbs or woody plants; leaves mostly compounds, alternate and with stipules; flowers perfect, irregular and zygomorphic; calyx of 4 or 5 more or less united sepals; petals 5, the upper, called the standard or banner, enlarged and enclosing the others in the bud, the two lowermost united to form the keel and enclose the pistil and stamens, the two lateral form the wings; stamens usually 10, and diadelphious, 9 being united by their filaments, the other being free; ovary superior, 1- or sometimes 2-celled by intrusion of the sutures; ovules 1-many; fruit a legume, or a loment by constriction between the seeds. Members of this family are usually known as legumes.

## 1A. Leaflets 3.

1B. Flowers in dense heads..... 1. *Trifolium*

2B. Flowers not in dense heads.

1C. Pods rugose, ovoid..... 2. *Melilotus*

2C. Pods coiled or curved..... 3. *Medicago*

## 2A. Leaflets more than 3.

1B. Leaves palmately compound.

1C. Leaflets serrulate..... 1. *Trifolium*

2C. Leaflets entire..... 4. *Lupinus*

2B. Leaves pinnately compound.

1C. Leaves usually with tendrils.

1D. Styles filiform with a tuft or ring of hairs at the apex..... 8. *Vicia*

2D. Styles flattened upward, hairy down inner side.... 9. *Lathyrus*

2C. Leaves without tendrils.

1D. Fruit a loment..... 7. *Hedysarum*

2D. Fruit on ordinary pod.

1E. Keel of the corolla acute or subulate at the apex. 6. *Oxytropis*

2E. Keel of the corolla obtuse at apex..... 5. *Astragalus*



## TRIFOLIUM (Tourn.) L.

Herbs; leaves denticulate; flowers white, pink, purple, red, or yellow, in dense heads or spikes; calyx pedicelled, with 5 subulate teeth; corolla persistent, the wings narrow and longer than the keel; pod flattened or terete, included in the persistent corolla, 1- to 6-seeded. (Latin three, and leaf.) With the exception of No. 8 all the species are introduced and only Nos. 4, 5, and 6 are common.

- |  |                            |
|--|----------------------------|
| 1A. Leaves mostly 5-foliolate.....             | 1. <i>T. lupinaster</i>    |
| 2A. Leaves trifoliolate.                       |                            |
| 1B. Heads involucre.                           |                            |
| 1C. Involucre cup-shaped.....                  | 7. <i>T. microcephalum</i> |
| 2C. Involucre rotate.                          |                            |
| 1D. Perennial, corolla 12 mm. long.....        | 8. <i>T. fimbriatum</i>    |
| 2D. Annual, corolla 6-8 mm. long.....          | 9. <i>T. variegatum</i>    |
| 2B. Heads without an involucre.                |                            |
| 1C. Annuals, flowers yellow.                   |                            |
| 1D. Heads 10- to 20-flowered.....              | 3. <i>T. dubium</i>        |
| 2D. Heads 20- to 40-flowered.....              | 2. <i>T. procumbens</i>    |
| 2C. Biennials or perennials.                   |                            |
| 1D. Peduncles terminal or subterminal.....     | 4. <i>T. pratense</i>      |
| 2D. Peduncles axillary.                        |                            |
| 1E. Stems prostrate, rooting at the nodes..... | 5. <i>T. repens</i>        |
| 2E. Stems ascending.....                       | 6. <i>T. hybridum</i>      |

1. *T. lupinaster* L.

## Lupine Clover

Perennial; stems erect or ascending, appressed pubescent, 3-5 dm. tall; leaflets linear-elliptic, acute, finely setose-serrulate, 2-4 cm. long; heads about 3 cm. thick; calyx pubescent, the tube about 3 mm. long, the teeth 5-8 mm. long; corolla pink, about 15 mm. long.

Escaped near Fairbanks and along the Yukon. Native of Eurasia. Fig. 690.

2. *T. procumbens* L.

## Low Hop Clover

Stems decumbent, 15-50 cm. long; leaflets obovate, cuneate at the base, rounded, truncate or emarginate at the apex, denticulate toward the apex, 10-15 mm. long, the terminal one stalked; flowers yellow, reflexed and brown in age, the standard broad and striate.

Native of Europe.

3. *T. dubium* L.

## Shamrock

Similar to *T. procumbens* but the leaflets are more distinctly cuneate, the standard narrower and only faintly striate and the whole plant more slender. This is claimed to be the true Shamrock.

Native of Europe.

4. *T. pratense* L.

## Red Clover

Stems more or less pubescent, branching, ascending, 2-7 dm. tall; stipules strongly veined, subulate-tipped; leaflets rounded or retuse at apex, minutely denticulate, 2-5 cm. long, often with dark spot near middle; heads ovoid, usually sessile; flowers rose-red, about 12 mm. long; calyx long-hairy.

Extensively naturalized, native of Eurasia.

5. *T. repens* L.

White or Dutch Clover

Stems creeping, glabrous; leaves long-petioled; stipules small, membranous, acute; leaflets broadly obovate, more or less emarginate at the apex, 8-25 mm. long; heads long-peduncled; flowers pedicelled, 8-12 mm. long, reflexed in fruit.

Extensively naturalized, native of Europe. Fig. 691.

6. *T. hybridum* L.

Alsike Clover

Perennial; stems erect or ascending, 2-7 dm. tall; heads long-peduncled; flowers pink to nearly white, pedicelled and reflexed in fruit; calyx teeth subulate. This is a natural species and not a hybrid as the name would indicate.

Extensively naturalized, native of Europe. Fig. 692.

7. *T. microcephalum* Pursh.

Small-headed Clover

Annual, stem sparingly villous, branched from the base, 2-4 dm. long; leaflets 5-15 mm. long, obcordate or cuneate-obovate, emarginate, serrate; involucre lobes 7-10, with scarious web-like margins; heads 5-10 mm. long; calyx pubescent, corolla rose to white, 6 mm. long.

Manley Hot Springs, B. C.—Mont.—Lower Calif.

8. *T. fimbriatum* Lindl.

Coast or Cow Clover

With slender, creeping rootstocks; stems decumbent, branching from the base, 1-4 dm. long; leaflets obovate to oblanceolate, finely setose-serrulate, 10-25 mm. long; involucre about 15 mm. broad deeply and lacinately lobed; heads 2-3 cm. broad; corolla about 12 mm. long, white or light purple, the wings reddish-purple.

Loring, B. C.—Calif.<sup>9</sup>

9. *T. variegatum* Nutt.

White-tipped Clover

Stems glabrous, decumbent or ascending, 2-10 dm. long; leaflets variable, the lower small, cuneate, obcordate, the upper obovate or oblong-lanceolate, 5-15 mm. long, setose-serrulate; heads 6-12 mm. broad; involucre lobed and deeply laciniate-toothed; corolla purple, white-tipped, 6-8 mm. long.

St. Michael, B. C.—Calif.

## 2. MELILOTUS (Tourn.) Hill

Our species sweet-scented herbs; flowers borne in spike-like racemes; calyx teeth nearly equal; pod ovoid, short and thick, indehiscent or nearly so. (Greek, honey and lotus.) Both species have become established at Fairbanks and Palmer in Alaska and at Mayo in Yukon. They are native to Eurasia.

Flowers white.....	1. <i>M. alba</i>
Flowers yellow.....	2. <i>M. officinalis</i>

1. *M. alba* Desv.

## White Sweet Clover

Stems erect, branched, 3–8 dm. tall; leaflets narrowly oblong- obovate, denticulate, 15–25 mm. long, narrowed at the base; flowers numerous, 4–6 mm. long; pod about 3 mm. long. Fig. 693a.

2. *M. officinalis* (L.) Lam.

## Yellow Sweet Clover

Similar to the preceding; leaflets somewhat broader and more sharply denticulate. Fig. 693b.

3. **MEDICAGO** (Tourn.) L.

Herbs with yellow or purple flowers in axillary heads or racemes; leaflets toothed; calyx with slender nearly equal lobes; pods curved or spirally coiled, in some species spiny. (Greek, from Medea.) Our species are escapes from cultivation or introduced weeds and are not common.

- |                               |                       |
|-------------------------------|-----------------------|
| 1A. Flowers purple.....       | 1. <i>M. sativa</i>   |
| 2A. Flowers yellow.....       |                       |
| 1B. Pods simply twisted.....  | 2. <i>M. falcata</i>  |
| 2B. Pods reniform.....        | 3. <i>M. lupulina</i> |
| 3B. Pods spirally coiled..... | 4. <i>M. hispida</i>  |

1. *M. sativa* L.

## Alfalfa

Perennial; much branched, partly decumbent or ascending; leaflets oblanceolate, truncate or retuse and toothed at the apex, 1–3 cm. long; corolla 8–10 mm. long; pod pubescent, spirally twisted into 2 or 3 coils.

Native of Europe. Fig. 694a.

2. *M. falcata* L.

## Yellow-flowered Alfalfa

Branched, decumbent or ascending perennial, 3–5 dm. tall; leaflets obovate-cuneate, toothed at the rounded, mucronate apex, 7–20 mm. long; flowers 7–10 mm. long; pod nearly straight but twisted, reticulated and finely pubescent, about 12 mm. long.

Near Fairbanks, native of Europe. Fig. 694b.

3. *M. lupulina* L.

## Nonsuch. Hop Clover

Annual, branched from the base, the branches decumbent or spreading, more or less pubescent throughout; leaflets cuneate, rounded, toothed, notched, mucronulate at the apex, the nerves ending in teeth; flowers in small head-like racemes, about 3 mm. long; pod pubescent, reticulated.

Occasionally adventitive in our area, native of Eurasia. Fig. 694c.

4. *M. hispida* Gaertn.

## Burr Clover

Annual; stems glabrous or with a few appressed hairs, branched from the base, spreading or ascending, 2–8 dm. long; leaflets obovate or obcordate, 8–20 × 5–15 mm., crenulate; pods coiled, reticulate, armed on the edges with hooked prickles.

Native of Eurasia.

## 4. LUPINUS (Tourn.) L.

Our species all perennials; flowers showy, in terminal racemes; leaves 5- to 15-foliate; calyx 2-lipped, the upper lip of 2 partly and the lower of 3 partly or wholly united sepals; corolla in ours blue, rarely white, often tinted with other colors; standard broad with reflexed margins; wings curved; keel sickle-shaped; stamens monodelphous; anthers alternately oblong and roundish; pod a flat 2-valved legume. (Latin, *Lupus*, a wolf.)

1A. Leaves green, thinly pubescent or glabrous above.

1B. Leaflets 9-15..... 3. *L. polyphyllus*

2B. Leaflets 5-10.

1C. Leaflets with acute tips..... 1. *L. arcticus*

2C. Leaflets with rounded tips..... 2. *L. nootkatensis*

2A. Leaflets canescent on both sides.

1B. Flowers subsessile..... 4. *L. lepidus*

2B. Flowers with pedicels 4-7 mm. long..... 5. *L. sericeus*

1. *L. arcticus* Wats.

## Arctic Lupine

Stems in clumps, 2-5 dm. tall; leaflets narrowly oblanceolate or linear obovate, appressed pubescent beneath, acute, often mucronate, 2-8 cm. long; stipules subulate; flowers often shaded pink or white, 15-18 mm. long; wings and standard nearly equal; calyx villous, the upper lip gibbous, 5-6 mm. long; lower lip 7-8 mm. long; pods with brown pubescence; seed brown, mottled. A variable group from which forms have been described as species.

Bering Sea—Arctic Archipelago—B. C. Fig. 695.

2. *L. nootkatensis* Donn.

## Nootka Lupine

Stems clustered, branched, varying from glabrous to densely villous, 2-10 dm. tall; leaflets 6-8, obovate or oblanceolate, 2-6 cm. long; racemes rather dense, up to 25 cm. long; flowers often shaded pink or white, rarely pure white, 13-18 mm. long; upper lip of calyx 8 mm. long, the lower lip 10 mm. long; wings and standard subequal; pod 3-4 cm. long. *L. kiskensis* C. P. Smith appears to be a very depauperate form of this species.

Mostly along the coast but extending to the Alaska Range, Attu Island to Vancouver Island. Fig. 696.

3. *L. polyphyllus* Lindl.

## Large-leaved Lupine

Stems stout, 6-15 dm. tall; leaflets narrowly oblanceolate, appressed-pubescent beneath, acute, 6-12 cm. long; racemes up to 50 cm. long; calyx gibbous on upper side, upper lip 4-5 mm. long, lower lip 5-6 mm. long; corolla blue, purple or reddish-purple; wings longer than the standard, about 15 mm. long; pods densely pubescent with long brown hairs.

Seward to Mt. McKinley Park, Vancouver Island—Mont.—Calif. Fig. 697.

4. *L. lepidus* Dougl.

## Prairie Lupine

Stems somewhat decumbent at base, densely silky, 15-40 cm. tall; leaves long-petioled; leaflets 5-9, oblanceolate, 12-25 mm. long, usually

folded; racemes 8–16 cm. long; flowers 10–13 mm. long; pods silky, 1–2 cm. long.

South Yukon—Hyder—Idaho—Calif. Fig. 698.

5. *L. sericeus* Pursh.

Silky Lupine

Appressed silky throughout; stems erect, 3–6 dm. tall; leaflets 5–9, oblanceolate, acute, 3–8 cm. long; racemes up to 15 cm. long; flowers about 1 cm. long; pod 2–3 cm. long, yellow.

Whitehorse, B. C.—Mont.—S. Dak.—Ore. Fig. 699.

5. ASTRAGALUS (Tourn.) L.

Herbs, ours all perennial and with evident stems; leaves usually odd-pinnate; flowers violet-purple, white or yellowish, borne in spikes or racemes; calyx tubular with nearly equal teeth; petals clawed, the standard erect, the keel blunt, about equaling the wings. (The Greek name of some legume.) In addition to the species here described, forms have been collected that may represent undescribed species.

- |  |                            |
|--|----------------------------|
| 1A. Pod sickle-shaped.....   | 1. <i>A. nutzotinensis</i> |
| 2A. Pod straight or nearly so.                                     |                            |
| 1B. Pod wholly 1-celled.   |                            |
| 1C. Pod compressed laterally.                                      |                            |
| 1D. Pod glabrous.....  | 2. <i>A. tenellus</i>      |
| 2D. Pod with black hairs.....                                      | 3. <i>A. amblyodon</i>     |
| 2C. Pod slightly or not at all compressed.                         |                            |
| 1D. Pod stipitate.   |                            |
| 1E. Pod glabrous.....  | 4. <i>A. americanus</i>    |
| 2E. Pod black-hairy.....   | 5. <i>A. umbellatus</i>    |
| 2D. Pod sessile.   |                            |
| 1E. Pod more than 15 mm. long.....                                 | 6. <i>A. polaris</i>       |
| 2E. Pod less than 1 cm. long.....                                  | 7. <i>A. yukonis</i>       |
| 2B. Pods with the lower suture inflexed.                           |                            |
| 1C. Septum incomplete.   |                            |
| 1D. Pod not sulcate on lower suture, both sutures prominent.       |                            |
| 1E. Pod compressed, nearly glabrous.....                           | 8. <i>A. aboriginorum</i>  |
| 2E. Pod more turgid, black-hairy.                                  |                            |
| 1F. Pod subsessile.....  | 9. <i>A. eucosmus</i>      |
| 2F. Pod stipitate.   |                            |
| 1G. Corolla 8–12 mm. long.....                                     | 10. <i>A. macounii</i>     |
| 2G. Corolla 12–15 mm. long.....                                    | 11. <i>A. harringtonii</i> |
| 2D. Pod sulcate on lower suture.                                   |                            |
| 1E. Pod erect or ascending.....                                    | 12. <i>A. williamsii</i>   |
| 2E. Pod drooping, stipitate.....                                   | 13. <i>A. alpinus</i>      |
| 2C. Septum complete or nearly so.                                  |                            |
| 1D. Partition of pod complete.....                                 | 15. <i>A. hypoglottis</i>  |
| 2D. Partition of pod not completely joined with upper suture ..... | 14. <i>A. vicifolius</i>   |

1. *A. nutzotinensis* Rousseau.

Sickle-pod Milk Vetch

*A. falciferous* Hult.

*Gynophoraria falcata* Rydb.

Stems weak and trailing, 3–20 cm. long; leaflets 9–19, obovate, elliptic or ovate, glabrate above, hirsute beneath; peduncles 1- to 4-flowered; flowers tinted lilac, 12–15 mm. long; calyx minutely black-hairy, the subulate teeth nearly as long as the tube; pod minutely black-hairy, 3–5 cm. long.

Chickaloon—Mt. McKinley Park—Yukon. Fig. 700.

2. *A. tenellus* Pursh. Loose-flowered Milk Vetch  
*Homalobus tenellus* (Pursh) Britt.

Caespitose; stems 3–5 dm. tall, sparingly strigose; leaflets 11–21, linear or oblong, obtuse at the apex, 1–2 cm. long, 1–3.5 mm. wide, glabrous on both sides or with a few hairs beneath; racemes several- to many-flowered; calyx-tube about 2 mm. long, the teeth slightly shorter; corolla ocreoleucous, 6–10 mm. long; pod stipitate,  $8-10 \times 3$  mm., reticulate.

Yukon—lower Mackenzie—Manitoba—Colo.—Nev. Fig. 701.

3. *A. amblyodon* Kearney.  
*Homalobus amblyodon* (Kearney) Rydb.

Stems caespitose, decumbent or prostrate, 1 dm. or less long; leaflets 5–13, oval or obovate, retuse at the apex, glabrous above, sparingly strigose beneath, 3–5 mm. long; peduncles few-flowered; calyx black-hairy, the tube about 3 mm. long, the teeth scarcely 1 mm. long; corolla, 10–12 mm. long.

Alaska Penin. and Mt. McKinley Park.

4. *A. americanus* (Hook.) M. E. Jones. Arctic Milk Vetch  
*Phaca americana* (Hook.) Rydb.

Erect, 3–10 dm. tall, glabrous below, slightly pubescent above; leaflets oval, elliptic or oblong, obtuse, glabrous above, somewhat pubescent beneath, 2–5 cm. long; calyx about 4 mm. long, nearly glabrous, the margin ciliate, the teeth short; pod glabrous, its stipe about 5 mm. long, the body about 2 cm. long.

Central Alaska—Great Slave Lake—Que.—Wyo.—B. C. Fig. 702.

5. *A. umbellatus* Bunge. Hairy Arctic Milk Vetch  
*A. littoralis* (Hook.) Cov. & Kearney.  
*Phaca littoralis* (Hook.) Rydb.

Stems more or less pubescent, 5–25 cm. tall; leaflets 7 or 9, oblong to ovate, glabrous above, pubescent beneath, 12–25 mm. long; peduncles 5- to 15-flowered; flowers yellowish, about 15 mm. long; calyx 5–8 mm. long, the teeth triangular, short, pubescent; pod short-stipitate, 15–20 mm. long, covered with short, black pubescence.

Alaska and Yukon except the southeastern coast, Eurasia. Fig. 703.

6. *A. polaris* (Seem.) Benth. Polar Milk Vetch  
*Phaca polaris* (Seem.) Rydb.

Stems slender, decumbent or creeping, 1–8 cm. long; leaflets 11–15, ovate or obovate,  $3-10 \times 3-5$  mm., notched at the apex; racemes 1- to 5-flowered; calyx black-hairy, the teeth triangular; corolla purple, about 15 mm. long; pod minutely strigulose, inflated, membranous,  $20-30 \times 10-15$  mm.

Cape Vancouver—Point Hope—Wiseman. Fig. 704.

7. *A. yukonis* M. E. Jones. Yukon Milk Vetch

Stems very slender, decumbent or ascending, 1-3 dm. long; leaflets 7-15, 4-12  $\times$  1.5-3.5 mm., glabrous above, strigose beneath; flowers 7-10 mm. long, the tips light purple, calyx black-hairy, the tube about 2.5 mm. long, the subulate teeth 1.5 mm. long; pod black-hairy, 5-7 mm. long.

Central Alaska—Yukon—western Mackenzie. Fig. 705.

8. *A. aboriginorum* Rich. Indian Milk Vetch

*A. linearis* (Rydb.) Pors.

*Atelophragma aboriginum* (Rich.) Rydb.

Caespitose, stems erect or decumbent at the base, 15-40 cm. tall; leaflets 9-13, oblong, lance-oblong or linear, more or less villous beneath, villous to glabrate above, 8-20 mm. long; peduncles longer than the leaves; racemes short in anthesis, elongated and lax in fruit; calyx black-hairy, the teeth subulate, nearly equaling the tube; corolla 8-10 mm. long, white, tinged with violet; pods long-stipitate, glabrous when mature, the body 15-25 mm. long, acute at both ends. A variety with black-strigulose pods is var. *muriei* Hult.

Seward Penin.—the Arctic Archipelago—Black Hills—Colo.—Nev. and the Gaspe Penin., Que. Fig. 706.

9. *A. eucosmus* Robin. Pretty Milk Vetch

*Atelophragma elegans* (Hook.) Rydb.

Stems glabrous or nearly so, somewhat branched, 25-55 cm. tall; leaflets usually 13 or 15, oblong or linear-oblong, 10-25 mm. long, glabrous above, strigose beneath; corolla about 8 mm. long, purple; pod usually black-hairy but sometimes white-hairy.

Seward Penin.—Great Bear Lake—Labr.—Newf.—Colo.—B. C. Fig. 707.

10. *A. macounii* Rydb. Macoun Milk Vetch

*Atelophragma collieri* Rydb. in part.

Stems 3-6 dm. tall, branched, somewhat angled, glabrous or nearly so; leaflets 11-19, elliptic or ovate, 15-30  $\times$  5-10 mm., glabrous and dark green above, paler and sparingly pilose beneath; calyx black-hairy, the teeth subulate and much shorter than the tube; corolla about 12 mm. long, nearly white; pods about 2 cm. long with a stipe about 5 mm. long, acute at each end.

Kobuk River—Great Bear Lake—Idaho—Colo. Fig. 708.

11. *A. harringtonii* Cov. & Standl. Harrington Milk Vetch

Stems branching, 2-5 dm. tall; leaflets 9-15, oblong, elliptic, or ovate, obtuse at the apex, glabrous or sparingly pubescent above, decidedly pubescent beneath, 1-4 cm. long; calyx pubescent with black hairs, the subulate teeth about the same length as the tube; pods about 15 mm. long, covered with mostly black pubescence.

Coastal region of Alaska north to Deering. Fig. 709.

12. *A. williamsii* Rydb.

Williams Milk Vetch

*Atelophragma williamsii* Rydb.

Stems ascending or erect, 3–6 dm. tall, more or less 4-angled; leaflets 9–13, oval to linear,  $15\text{--}35 \times 4\text{--}12$  mm., obtuse or retuse at the apex, glabrous or nearly so; racemes compact but elongating in fruit; calyx black-hairy, the tube about 3 mm. long, the teeth short; petals ochroleucous, the keel purplish-tipped; pods erect, subsessile, 10–14 mm. long, in age glabrous and reticulate, deeply sulcate on lower suture.

Central Alaska—Yukon. Fig. 710.

13. *A. alpinus* L.

Alpine Milk Vetch

Ascending or decumbent, branched, 1–4 dm. tall; leaflets 11–29, ovate, elliptic or obovate, glabrate above, pilose beneath, 6–15 mm. long; flowers violet, about 12 mm. long; calyx black-hairy, the teeth triangular-subulate, nearly as long as the tube; pod stipitate, densely black-hairy. A variable and widely distributed species occurring in several forms or races.

Circumpolar, south to Vt.—Colo.—Idaho. Fig. 711.

14. *A. vicifolius* Hult.

Vetch-leaved Milk Vetch

Caespitose; stems 15–50 cm. tall, angled, strigose; leaflets 13–17, linear-oblong,  $10\text{--}30 \times 2\text{--}6$  mm., strigose pubescent beneath, usually glabrous above; racemes dense, many-flowered; calyx pubescent with black hairs usually with some white ones intermixed, the tube 4–5 mm. long, the teeth short; corolla about 15 mm. long, ochroleucous with purple tip; pod white-hairy, about 6 mm. long.

Central Alaska—Yukon. Fig. 712.

15. *A. hypoglottis* L.

Purple Milk Vetch

*A. agrestis* Dougl.*A. tarletonis* Rydb.

Caespitose; stems branched, angled, decumbent or ascending, 1–3 dm. tall; leaflets 15–25, oblong to elliptic, emarginate, 6–15 mm. long, rather densely pubescent beneath, less so above; flowers purple, in dense heads; calyx black-pubescent; pods short, sessile, densely pilose.

Yukon—Great Slave Lake—Hudson Bay—Minn.—Calif. Eurasia. Fig. 713.

## 6. OXYTROPIS DC.

Tufted perennial, nearly acaulescent herbs resembling *Astragalus*; leaves odd-pinnate; flowers racemose or spicate, sometimes reduced to 1; calyx teeth nearly equal; petals clawed, the keel erect, its apex mucronate, acuminate, or appendaged; pod 2-valved, 1-celled or more often 2-celled by the intrusion of the ventral suture. (Greek, sharp keel.) A very critical and confusing group, forms occurring that can scarcely be assigned to any of the following species.



- 1A. Leaves unifoliate or trifoliate..... 3. *O. mertensiana*  
 2A. Leaves pinnate.  
   1B. Calyx lobes glandular.  
     1C. Stipules long-ciliate on the margins..... 4. *O. leucantha*  
     2C. Stipules also pubescent on the back.  
       1D. Calyx mostly white-hairy, pods abruptly pointed.. 5. *O. viscida*  
       2D. Calyx mostly black-hairy, pods more long-acuminate ..... 6. *O. viscidula*  
   2B. Calyx lobes not glandular.  
     1C. Peduncles rarely more than 2-flowered.  
       1D. Old stipules stiff, castaneous..... 7. *O. kokrinensis*  
       2D. Old stipules membranous and thin, light- or grayish-brown ..... 8. *O. nigrescens*  
     2C. Peduncles 2- to 5-flowered..... 9. *O. scammaniana*  
     3C. Peduncles mostly more than 5-flowered.  
       1D. Flowers blue or purplish.  
         1E. Pods reflexed.  
           1F. Racemes strongly elongated in fruit..... 1. *O. deflexa*  
           2F. Racemes short and head-like..... 2. *O. foliolosa*  
         2E. Pods ascending.  
           1F. Scapes 5- to 8-flowered.....10. *O. roaldi*  
           2F. Scapes up to 15-flowered.....11. *O. ?erecta*  
       2D. Flowers yellowish.  
         1E. Old stipules dark castaneous brown.....13. *O. maydelliana*  
         2E. Old stipules yellowish or light brown.....13. *O. gracilis*  
 3A. Leaves with verticillate leaflets (see also 11. *O. ?erecta*).  
   1B. Flowers blue.....14. *O. splendens*  
   2B. Flowers yellowish.....15. *O. varians*

1. *O. deflexa* (Pall.) A.DC.

## Deflexed-podded Oxytrope

*O. retrorsa* Fern.

Silky-pubescent, with short stems; peduncles 15–40 cm. tall; leaflets 23–45, crowded, lanceolate or ovate, silky, rounded at the base, acute at the apex, 5–15 mm. long; fruiting racemes 4–12 cm. long; flowers dingy white with bluish apex, 6–9 mm. long; calyx teeth subulate, about as long as the tube; pod pubescent with soft white or brown hairs, nearly 15 mm. long, strongly deflexed and the ventral suture deeply intruded.

Circumpolar, south to the Black Hills, N. Mex., Idaho, and B. C. Fig. 714.

2. *O. foliolosa* Hook.

## Foliose Oxytrope

Resembling *O. deflexa* but acaulescent; leaflets 15–29, appressed pilose, ovate, 2–10 mm. long; spike compact, 1–3 cm. long, 2- to 10-flowered; calyx campanulate, black-pilose, the lance-subulate lobes about equaling the tube; corolla deep violet, 8–10 mm. long; pod stipitate within the calyx, 10–15 mm. long, black-hirsute.

Yukon—Hudson Str.—Newf.—Colo.

3. *O. mertensiana* Turcz.

## Mertens Oxytrope

Less than 1 dm. tall; leaves usually reduced to 1 leaflet but sometimes trifoliate; leaflets linear-elliptic, ciliate on the margins, acute at both ends, 15–30 mm. long; peduncles 1- to 3-flowered, more or less villous; flowers purple, about 14 mm. long; calyx black-wooly, about 15 mm. long, short-beaked.

Central and northwest Alaska, northeast Asia. Fig. 715.

4. *O. leucantha* (Pall.) Bunge.*O. borealis* Hook.

Caespitose, hirsute and glandular; leaflets 17–25, the margins revolute, upper surface glabrous, lower surface ciliate, 5–10 mm. long; peduncles 5–30 cm. tall; heads short and compact, usually 6- to 12-flowered; calyx densely black and white long-hairy, the tube about 6 mm. long, the teeth 3–4 mm. long; corolla violet blue, about 15 mm. long; pod black-hairy.

Eastern Asia and Bering Sea region—western Yukon. Fig. 716.

5. *O. viscida* Nutt.

Vicid Oxytrope

*Aragallus viscidus* (Nutt.) Greene.

Caespitose; leaflets up to 57 in number, usually acute, 3–18 mm. long; scapes 8–30 cm. tall, erect, hirsute; spikes 3–8 cm. long; calyx villous, the tube about 5 mm. long, the teeth 3 mm. long; corolla violet or whitish, about 12 mm. long; pod minutely pubescent, 12–15 mm. long.

Central Alaska—Mont.—Colo.—Nev. Fig. 717.

6. *O. viscidula* (Rydb.) Tidestrom.*Aragallus viscidulus* Rydb.

Caespitose; leaflets 17–31, 5–13 mm. long, sparingly villous and glandular; scapes 6–22 mm. tall; spikes 2–5 cm. long; calyx soft-hairy, the tube 4–5 mm. long, nearly equaled by the teeth; corolla purple with yellowish base, about 12 mm. long; pod 10–15 mm. long, finely black-hairy.

Katmai—Wiseman—Great Slave Lake—Colo.—Utah. Fig. 718.

7. *O. kokrinensis* Pors.

Kokrines Mountains Oxytrope

Caudices long, densely covered by long-persisting, ferrugineous stipules with attached petioles; free part of stipules silky villous, in age merely ciliate to almost glabrous, long-triangular, acute; leaves long-petioled, 3–5 cm. long with 3 or 4 pairs of revolute leaflets, long, silky-villous; scapes barely exceeding the leaves, usually 2-flowered; calyx purplish-brown, villous, the teeth subulate, one half as long as the tube; corolla purple, 10–15 mm. long; pod stipitate within the calyx, 20–25 × 6–8 mm., with short grayish-black, appressed pubescence.

Kokrines Mountains.

8. *O. nigrescens* (Pall.) Fisch.

Blackish Oxytrope

Densely caespitose with branching caudex a few centimeters long; leaves and peduncles silky-canescens with white hairs, leaflets 7–13, ovate to lanceolate, 3–8 mm. long; stipules scarious, the lobes lanceolate and ciliate margined; scapes 1- or 2-flowered; calyx densely black-hairy, the lobes narrow; corolla blue or purplish, 15–25 mm. long; pods 2–3 cm. long, inflated. This species is represented in our area by 2 subspecies. Ssp. *bryophila* (Greene) Hult. (*Aragallus bryophilus* Greene) has the free part of the stipules long attenuate. Ssp. *pygmaea* (Pall.) Hult. (*O. pygmaea* (Pall.) Fern.) is more pulvinate in habit and has shorter, blunt stipules.

The type form Asiatic, ssp. *bryophila* in central and western Alaska, ssp. *pygmaea* in northern and central Alaska to Hudson Bay. Fig. 719.

9. *O. scammaniana* Hult.

Scamman Oxytrope

*O. arctica* Am. auct.

Densely tufted; leaves 15–40 mm. long; leaflets 7–19, 4–8 × 1–3 mm., hirsute, at least beneath, often ending in a tuft of hairs; scapes 2–7 cm. tall, 2- to 5-flowered; Calyx densely black-hairy, the tube 3–4 mm. long, the teeth 2–2.5 mm. long; corolla violet, 11–15 mm. long; pod black-hairy and with incurved tip, about 15 mm. long.

Central and eastern Alaska. Fig. 720.

10. *O. roaldi* Ostenf.

Roald Oxytrope

Loosely caespitose; free part of stipules deltoid, hyaline, pubescent on back with long, appressed, white or yellowish appressed hairs; leaves up to 9 cm. long; leaflets 11–19, 3–10 mm. long, serico-villous; scapes 1 dm. or less tall, 3- to 8-flowered; calyx black-hairy; corolla purplish, about 2 cm. long.

Rare, arctic Asia, Alaska and Yukon—Victoria Island and Coronation Gulf.

11. *O. ?erecta* Kom.

Stipules hairy, the free part long-attenuate, hyaline, reticulate; leaves up to 13 cm. long, pinnate or the leaflets verticillate; leaflets acute, 12–20 mm. long, about 3 mm. wide, sparingly silky on both sides; bracts linear-setaceous, up to 13 mm. long, white-hairy; calyx with long white and short black hairs, the tube 6–8 mm. long, the teeth about 5 mm. long; corolla violet, about 2 cm. long; ovary pubescent, slightly stipitate.

North slope of Brooks Range. *O. erecta* is a Kamchatka species and this may prove to be an unnamed form.

12. *O. maydelliana* Trautv.

Maydell Oxytrope

More or less villous-pubescent throughout with rather long, white hairs except the calyx on which the hairs are black or mixed; leaflets 11–19, ovate-lanceolate, 4–16 mm. long; scapes several- to many-flowered; inflorescence a bracted spike, often head-like; flowers yellowish, 12–18 mm. long; pod generally with both black and white hairs, 12–15 mm. long, tipped with the beak-like persistent style.

Eastern Asia—arctic Alaska—Baffin Land—Labr.—Alaska Penin. Fig. 721.

13. *O. gracilis* (A. Nels.) K. Schum.

Northern Yellow Oxytrope

*O. campestris* Am. auct.

Leaflets 21–31, more or less appressed-silky, usually acute, 10–25 mm. long; scapes 2–4 dm. tall; spikes 4–12 cm. long; calyx silky, the tube 6–8 mm. long, the linear-subulate teeth 2.5–4 mm. long; corolla yellowish, about 15 mm. long; pod black-hirsute, often with some white hairs, about 2 cm. long.

Central and south Alaska—Alta.—Man.—S. Dak.—Idaho. Fig. 722.

14. *O. splendens* Dougl. Showy Oxytrope  
*O. richardsonii* Hook.

Silky-villous throughout; leaflets 20–50, in whorls of 2–4, oblong-lanceolate, 8–20 mm. long; scapes 1–3 dm. tall; calyx densely villous, the teeth narrow, less than half the length of the tube; corolla violet blue, 10–15 mm. long; pod long-villous, long-pointed, about 15 mm. long.

East Alaska—Yukon—Sask.—Minn.—Mont.—B. C. Fig. 723.

15. *O. varians* (Rydb.) Hult. Variable Oxytrope  
*Aragallus varians* Rydb.

Leaflets 25–50, many of them in verticils, densely silky-villous when young, 1–2 cm. long; scapes 15–40 cm. tall; spikes 4–12 cm. long; calyx silky-villous, the tube about 5 mm. long, the teeth 2–3 mm. long; corolla about 12 mm. long, yellowish; pod villous and with short black hairs, 12–15 mm. long.

Eastern and south central Alaska to Yukon—Victoria Land—Great Bear Lake. Fig. 724.

#### 7. HEDYSARUM (Tourn.) L.

Perennials; flowers showy, in axillary racemes; calyx bracteolate and with 5 nearly equal teeth; standard obovate or obcordate, clawed; keel longer than the wings, obliquely truncate; pod flat, divided transversely into rounded or rhombic, 1 seeded internodes forming what is known as a loment.

Calyx teeth ovate, acute, shorter than the tube..... 1. *H. alpinum americanum*  
 Calyx teeth subulate, longer than the tube..... 2. *H. mackenzii*

1. *H. alpinum* L. ssp. *americanum* (Michx.) Fedtsch.

American Hedysarum

*H. boreale* Nutt.

*H. auriculatum* Eastw.

Stems erect or ascending, glabrous or nearly so, often strigose above, 2–7 dm. tall; leaflets 9–21, variable, often mucronulate, rounded at the base, 15–30 mm. long, sparingly hairy beneath; racemes long; flowers violet purple to white, numerous, deflexed, 12–18 mm. long; pod of 3–5 strongly reticulated, mostly oval joints 6–10 mm. long. The var. *grandiflorum* Rollins (*H. truncatum* Eastw.) has flowers longer than 16 mm.

The whole species circumpolar, ssp. *americanum* south to B. C., Wyo., S. Dak., N. Hamp. and Maine. Fig. 725.

2. *H. mackenzii* Rich.

Wild Sweet Pea

Minutely pubescent or strigose; stems 3–6 dm. tall; leaflets 9–17, elliptic, 1–3 cm. long, glabrate above, grayish strigose beneath; flowers fragrant, violet purple, 17–20 mm. long; pods normally about 6-jointed, minutely strigose and cross-reticulated, the internodes nearly orbicular, 5–7 mm. long.

North Asia—Banks and Victoria Lands—Man.—Alta—Ore. Also in Que. and Newf. Fig. 726.

## 8. VICIA L.

Climbing or trailing herbs; leaves pinnate, bearing tendrils at the tip; flowers axillary, solitary or more often borne in racemes; calyx somewhat oblique, 5-toothed, the upper 2 shorter; standard obovate or oblong, clawed; wings adherent to the keel; style slender, with a tuft or ring of hairs at the summit; pod flat, 2-valved, dehiscent, few- to several-seeded. (The classical Latin name.)

- 1A. Flowers solitary or in pairs..... 5. *V. angustifolia*  
 2A. Flowers in racemes.  
   1B. Racemes short, 2- to 8-flowered..... 1. *V. americana*  
   2B. Racemes elongated, many-flowered.  
     1C. Plant villous with spreading hairs..... 3. *V. villosa*  
     2C. Glabrous or with appressed pubescence.  
       1D. Flowers 9-12 mm. long..... 4. *V. cracca*  
       2D. Flowers more than 12 mm. long..... 2. *V. gigantea*

1. *V. americana* Muhl. American Vetch

Perennial; stems 6-10 dm. long, sparsely pubescent; leaflets 8-16, nearly elliptic, cuspidate and often with a few serrations, 15-45 mm. long; calyx teeth lanceolate; corolla purple, 15-18 mm. long; pod glabrous, 3-4 cm. long.

Central Alaska—Great Slave Lake—Dela.—Va.—Mo.—Texas—Ariz.—Calif. Fig. 727.

2. *V. gigantea* Hook. Sitka Vetch  
*V. sitchensis* Bong.

A vigorous perennial; stems slightly pubescent below, more so toward the summit; leaves 15-30 cm. long including the tendrils; leaflets 14-32, ovate-oblong, obtuse or rounded and mucronulate at the apex, 18-60 mm. long, joined to the rachis by short stalk; peduncles 5- to 16-flowered; flowers purple or ochroleucous, 12-16 mm. long; pod stipitate, glaucous, blackish, about 45 × 15 mm.

Along the coast, Cook Inlet—central Calif. Fig. 728.

3. *V. villosa* Roth. Hairy Vetch

Annual or biennial; stems villous with spreading hairs, up to 15 dm. long; leaflets linear to oblong-linear, 15-30 mm. long; flowers 15-20 mm. long; pod up to 3 mm. long.

Escaped from cultivation at Palmer. Native of Eurasia.

4. *V. cracca* L. Cow Vetch

Perennial; stems slender, usually finely pubescent, up to 1 m. long; leaflets 8-24, linear to lance-oblong, 12-20 mm. long; racemes elongated and densely-flowered; flowers violet; pods glabrous, 18-24 mm. long.

Escaped at Fairbanks and Palmer. Native of Eurasia.

5. *V. angustifolia* (L.) Reich.

Narrow-leaved Vetch

Stems slender, glabrous or puberulent, 3–6 dm. long; leaflets 4–16, 8–35 × 2–4 mm.; flowers 2 or more usually 1 in the upper axils, purple; pod linear, glabrous, 25–50 × 5–7 mm.

Introduced at Sitka. Native of Europe.

## 9. LATHYRUS (Tourn.) L.

Ours perennial herbaceous vines with horizontal rootstocks; leaves pinnate and tendril-bearing; flowers in racemes; calyx oblique or gibbous at the base, the teeth nearly equal or the upper shorter; standard obovate, emarginate and clawed; wings oblique, adherent to the shorter keel; stamen diadelphous above, monadelphous below; style curved, hairy along the inner side; pod linear, flattened, continuous between the seeds. (Ancient Greek name of some legume.)

1A. Stipules nearly as large as the leaflets..... 1. *L. maritimus*

2A. Stipules much smaller than the leaflets.

1B. Stems winged, leaflets narrow..... 2. *L. palustris pilosus*

2B. Stems simply keeled, leaflets wider..... 3. *L. venosus*

1. *L. maritimus* (L.) Bigel.

Beach Pea

*L. japonicus* Willd.

Stems glabrous or in northern forms somewhat pubescent, 2–6 dm. long; stipules ovate, sagittate-hastate, acute, 2–4 cm. long; leaflets 3–6 pairs, oblong-elliptical, obtuse and mucronate at apex; tendrils often branched; flowers purple, showy, 18–25 mm. long; pod linear-oblong, 4–5 cm. in length.

Near the sea, circumpolar and widely distributed. Fig. 729.

2. *L. palustris* L. ssp. *pilosus* (Cham.) Hult.

Wild Pea

Stems slender, somewhat pubescent, angled and winged, 4–10 dm. long; leaflets 2–4 pairs, 25–50 cm. long, 3–10 mm. wide, mucronate at the apex; tendrils mostly branched; peduncles 2- to 6-flowered; corolla purple, 14–20 mm. long; pods linear, slightly pubescent, 40–60 × 6 mm.

The whole species circumboreal. the ssp. south to N. Car. and Okla. Fig. 730.

3. *L. venosus* Muhl.

Veiny Pea

Stems slender, sparingly pubescent, 5–10 dm. long; leaflets 6–14, elliptic, 2–5 cm. long; stipule small, entire; tendrils well developed; peduncles about as long as the leaves, 5- to 10-flowered; calyx pubescent, the teeth shorter than the tube; corolla 15–18 mm. long, the standard purplish, the other petals whitish; pods glabrous, linear, 4–5 cm. long.

Hyder—Sask.—Ont.—Penn.—Ga.—La.—Kans.—Mont.

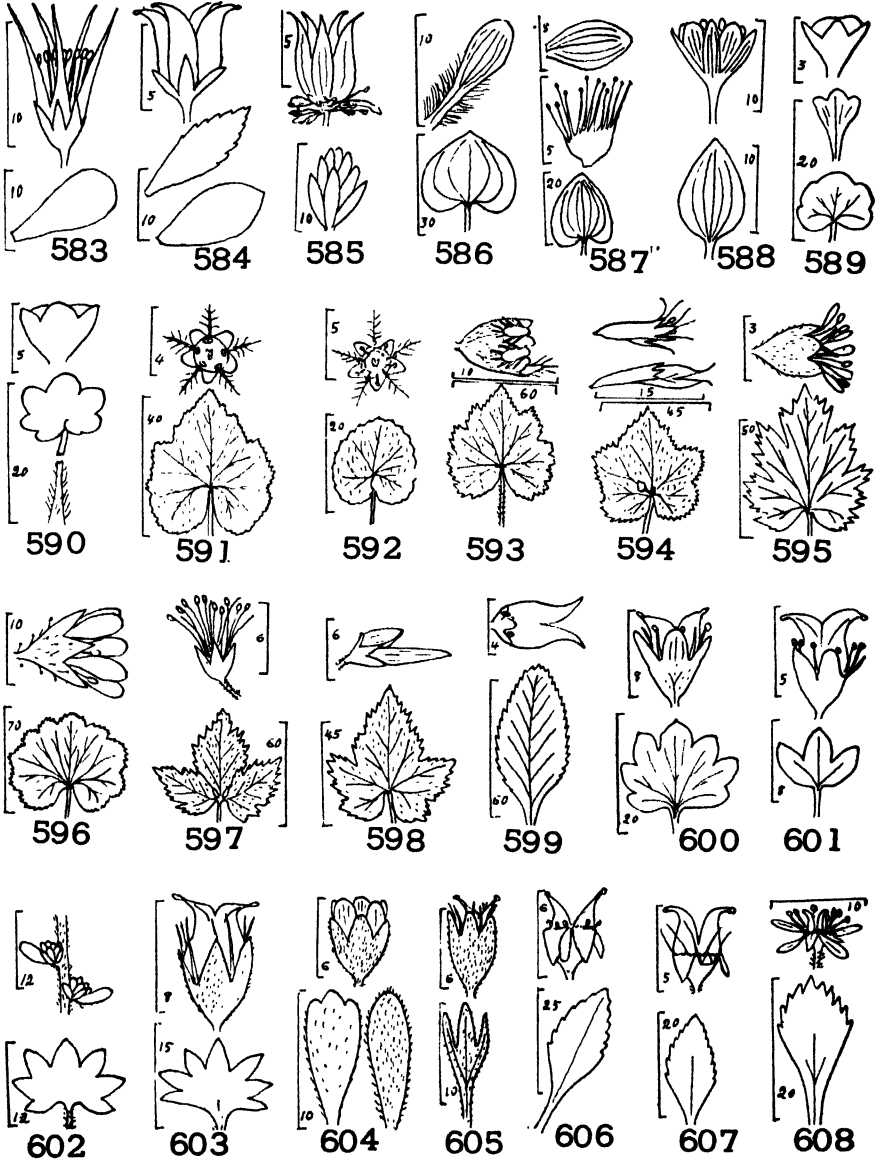
## PLATE XXIV

Scale marked in millimeters

FIG.

- 583. *Sedum oregonum* Nutt. Flower and leaf.
- 584. *Sedum roseum* (L.) Scop. Fruit and leaves.
- 585. *Sedum stenopetalum* Pursh. Fruit and cluster of leaves.
- 586. *Parnassia fimbriata* König. Petal and leaf.
- 587. *Parnassia palustris* L. Petal, staminodium, and leaf.
- 588. *Parnassia kotzebuei* C. & S. Flower and leaf.
- 589. *Chrysosplenium tetrandrum* Th. Fries. Fruit, bract, and leaf.
- 590. *Chrysosplenium wrightii* Franch. & Sav. Fruit, leaf, and base of petiole.
- 591. *Mitella pentandra* Hook. Flower and leaf.
- 592. *Mitella nuda* L. Flower and leaf.
- 593. *Tellima grandiflora* (Pursh) Dougl. Flower and leaf.
- 594. *Tolmiea menziesii* (Pursh) Torr. & Gray. Flower, fruit, and leaf.
- 595. *Heuchera glabra* Willd. Flower and leaf.
- 596. *Boykinia richardsonii* (Hook.) Gray. Flower and leaf.
- 597. *Tiarella trifoliata* L. Flower and leaf.
- 598. *Tiarella unifoliata* Hook. Fruit and leaf.
- 599. *Leptarrhena pyrolifolia* (D. Don) Sér. Fruit and leaf.
- 600. *Saxifraga bracteata* D. Don. Fruit and lower leaf.
- 601. *Saxifraga rivularis* L. Fruit and lower leaf.
- 602. *Saxifraga cernua* L. Stem bulblets and lower leaf.
- 603. *Saxifraga radiata* Small. Fruit and lower leaf.
- 604. *Saxifraga adscendens oregonensis* (Raf.) Bacigalupi. Flower and leaves.
- 605. *Saxifraga caespitosa sileniflora* (Sternb.) Hult. Fruit and leaf.
- 606. *Saxifraga hieracifolia* Wallst. & Kit. Fruit and leaf.
- 607. *Saxifraga nivalis* L. Fruit and leaf.
- 608. *Saxifraga davurica grandipetala* (Engl. & Irmscher) Hult. Flower and leaf.

PLATE XXIV





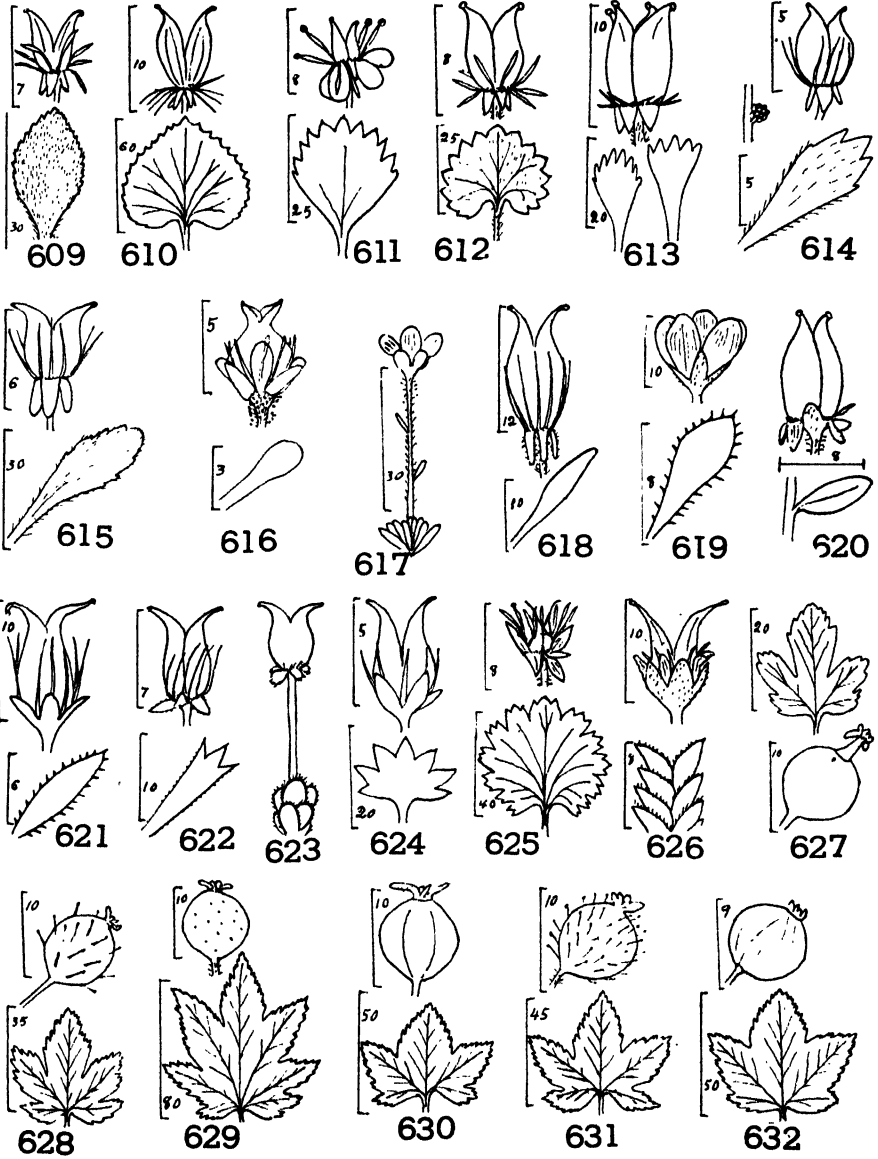
## PLATE XXV

Scale marked in millimeters

FIG.

- 609. *Saxifraga reflexa* Hook. Fruit and leaf.
- 610. *Saxifraga spicata* D. Don. Fruit and leaf.
- 611. *Saxifraga lyallii* Engler. Fruit and leaf.
- 612. *Saxifraga punctata nelsoniana* (D. Don) Hult. Fruit and leaf.
- 613. *Saxifraga unalaschkensis* Sternb. Fruit and leaves.
- 614. *Saxifraga foliolosa* R. Br. Fruit, bulblet, and leaf.
- 615. *Saxifraga ferruginea* Grah. Fruit and leaf.
- 616. *Saxifraga aleutica* Hult. Fruit and leaf.
- 617. *Saxifraga serpyllifolia* Pursh. Flowering plant.
- 618. *Saxifraga hirculus* L. Fruit and leaf.
- 619. *Saxifraga flagellaris* Willd. Flower and leaf.
- 620. *Saxifraga tolmiei* Torr. & Gray. Fruit and leaf.
- 621. *Saxifraga bronchialis funstonii* (Small) Hult. Fruit and leaf.
- 622. *Saxifraga tricuspidata* Retz. Fruit and leaf.
- 623. *Saxifraga eschscholtzii* Sternb. Flowering branch.
- 624. *Saxifraga nudicaulis* D. Don. Fruit and leaf.
- 625. *Saxifraga mertensiana* Bong. Flower and leaf.
- 626. *Saxifraga oppositifolia* L. Fruit and portion of stem.
- 627. *Ribes oxycanthoides* L. Leaf and fruit.
- 628. *Ribes lacustre* (Pers.) Poir. Fruit and leaf.
- 629. *Ribes bracteosum* Dougl. Fruit and leaf.
- 630. *Ribes hudsonianum* Rich. Fruit and leaf.
- 631. *Ribes laxiflorum* Pursh. Fruit and leaf.
- 632. *Ribes triste* Pall. Fruit and leaf.

PLATE XXV



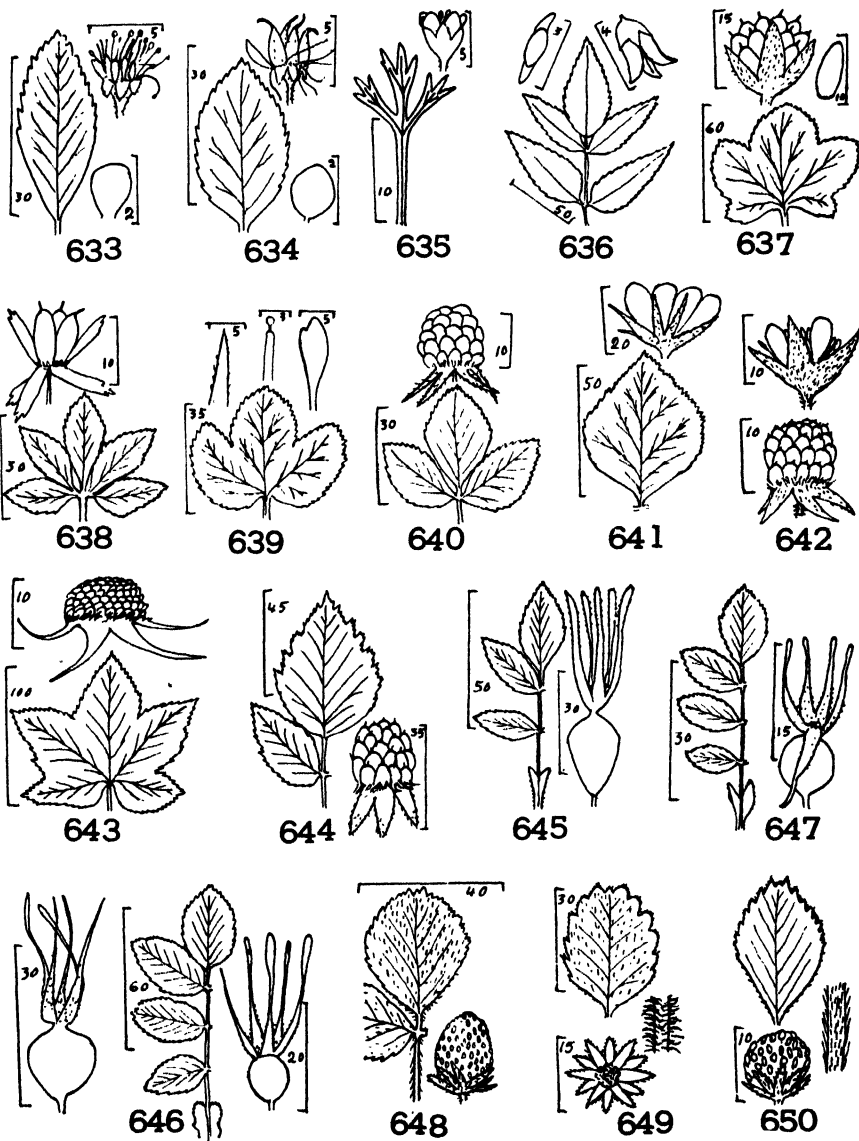
## PLATE XXVI

Scale marked in millimeters

FIG.

- 633. *Spiraea menziesii* Hook. Leaf, flower, and petal
- 634. *Spiraea beauverdiana* Schneid. Leaf, fruit, and petal.
- 635. *Luetkea pectinata* (Pursh) Kunze. Leaf and flower.
- 636. *Aruncus vulgaris* Raf. Seed, fruit, and part of leaf.
- 637. *Rubus chamaemorus* L. Fruit, druplet, and leaf.
- 638. *Rubus pedatus* Smith. Fruit and leaf.
- 639. *Rubus stellatus* Smith. Sepal, stamen, petal, and leaf.
- 640. *Rubus arcticus* L. Fruit and leaf.
- 641. *Rubus alaskensis* Bailey. Flower and leaflet.
- 642. *Rubus strigosus* Michx. Flower and fruit.
- 643. *Rubus parviflorus* Nutt. Fruit and leaf.
- 644. *Rubus spectabilis* Pursh. Part of leaf and fruit.
- 645. *Rosa acicularis* Lindl. Part of leaf and fruit.
- 646. *Rosa nutkana* Presl. Fruits and part of leaf.
- 647. *Rosa woodsii* Lindl. Part of leaf and fruit.
- 648. *Fragaria chiloensis* (L.) Duch. Part of leaf and fruit.
- 649. *Fragaria bracteata* Heller. Leaflet, calyx with bracts, and section of petiole.
- 650. *Fragaria glauca* (Wats.) Rydb. Leaflet, fruit, and section of petiole.

PLATE XXVI



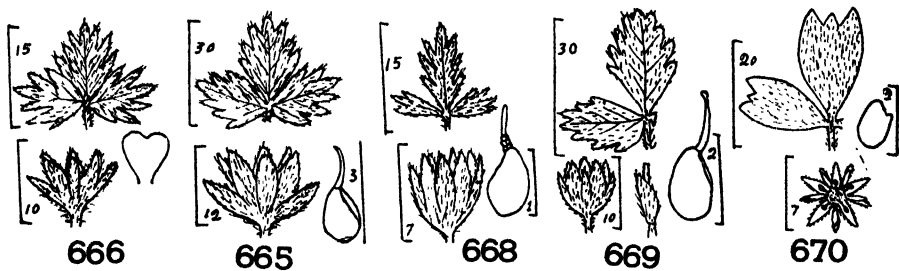
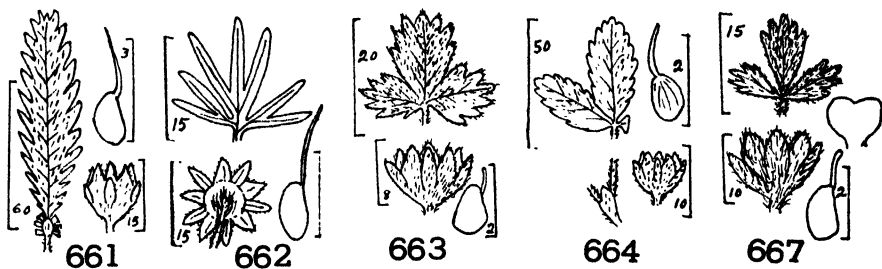
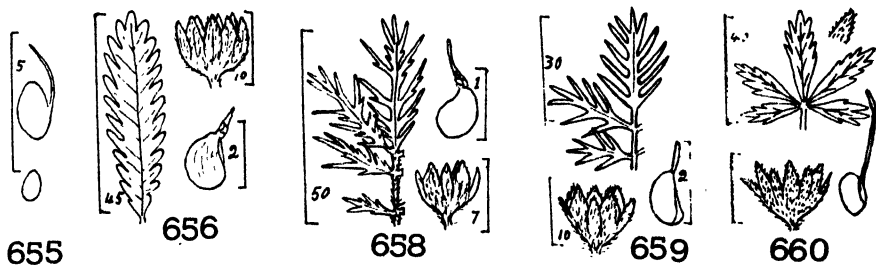
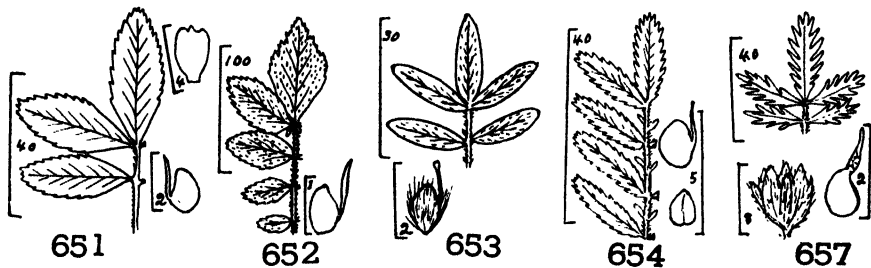
## PLATE XXVII

Scale marked in millimeters

Fig.

- 651. *Potentilla palustris* (L.) Scop. Part of leaf, petal, and achene.
- 652. *Potentilla arguta* Pursh. Part of leaf and achene.
- 653. *Potentilla fruticosa* L. Leaf and achene.
- 654. *Potentilla anserina* L. Part of leaf and achene.
- 655. *Potentilla pacifica* Howell. Achene.
- 656. *Potentilla pennsylvanica* L. Leaflet, calyx with bracts, and achene.
- 657. *Potentilla pectinata* Fisch. Leaf, calyx with bracts, and achene.
- 658. *Potentilla virgulata* A. Nels. Part of leaf, calyx with bracts, and achene.
- 659. *Potentilla multifida* L. Part of leaf, calyx with bracts, and achene.
- 660. *Potentilla diversifolia* Lehm. Leaf, calyx with bracts, and achene.
- 661. *Potentilla gracilis* Dougl. Leaflet, achene, and calyx with bracts.
- 662. *Potentilla biflora* Willd. Leaf, back of flower, and achene.
- 663. *Potentilla emarginata* Pursh. Leaf, calyx with bracts, and achene.
- 664. *Potentilla monspeliensis* L. Part of leaf, stipule, calyx with bracts, and achene.
- 665. *Potentilla villosa* Pall. Leaf, calyx with bracts, and achene.
- 666. *Potentilla uniflora* Ledeb. Leaf, calyx with bracts, and petal.
- 667. *Potentilla vahliana* Lehm. Leaf, calyx with bracts, petal, and achene.
- 668. *Potentilla hookeriana* Lehm. Leaf, calyx with bracts, and achene.
- 669. *Potentilla nivea* L. Part of leaf, calyx with bracts, stipule and achene.
- 670. *Sibbaldia procumbens*. Part of leaf, flower, and achene.

PLATE XXVII



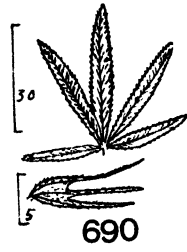
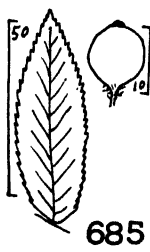
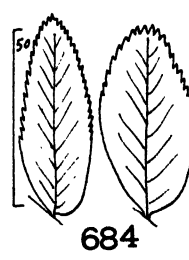
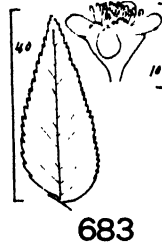
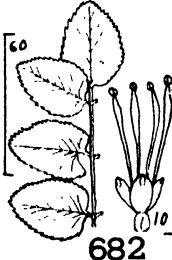
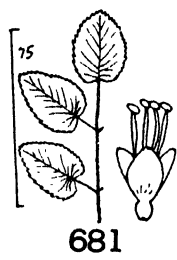
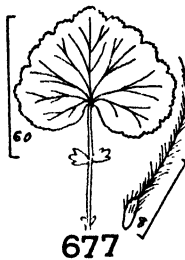
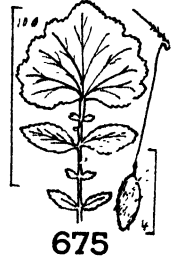
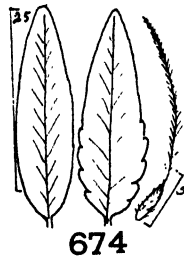
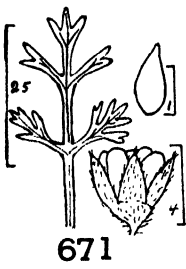
## PLATE XXVIII

Scale marked in millimeters

FIG.

- 671. *Chamaerhodos nuttallii* (T. & G.) Pickering. Leaf, achene, and flower.
- 672. *Dryas drummondii* Rich. Leaf and achene.
- 673. *Dryas octopetala* L. Leaf and achene.
- 674. *Dryas integrifolia* Vahl. Leaves and achene.
- 675. *Geum macrophyllum* Willd. Leaf and achene.
- 676. *Geum rossii* (R. Br.) Sér. Leaf and achene.
- 677. *Geum calthifolium* Menz. Leaf and achene.
- 678. *Geum pentapetalum* (L.) Makino. Leaf with stipules and achene.
- 679. *Geum glaciale* Adams. Leaf and achene.
- 680. *Sanguisorba officinalis* L. Part of leaf and flower.
- 681. *Sanguisorba menziesii* Rydb. Part of leaf and flower.
- 682. *Sanguisorba sitchensis* C.A.Mey. Part of leaf and flower.
- 683. *Sorbus sambucifolia* (C. & S.) Roem. Leaflet and flower.
- 684. *Sorbus sitchensis* Roem. Leaves.
- 685. *Sorbus scopulina* Greene. Leaflet and fruit.
- 686. *Malus fusca* (Raf.) Schneider. Leaves and fruit.
- 687. *Amelanchier alnifolia* Nutt. Leaf and flower.
- 688. *Amelanchier florida* Lindl. Leaf and fruit.
- 689. *Crataegus douglasii* Lindl. Leaves and fruit.
- 690. *Trifolium lupinaster* L. Leaf and calyx.

PLATE XXVIII





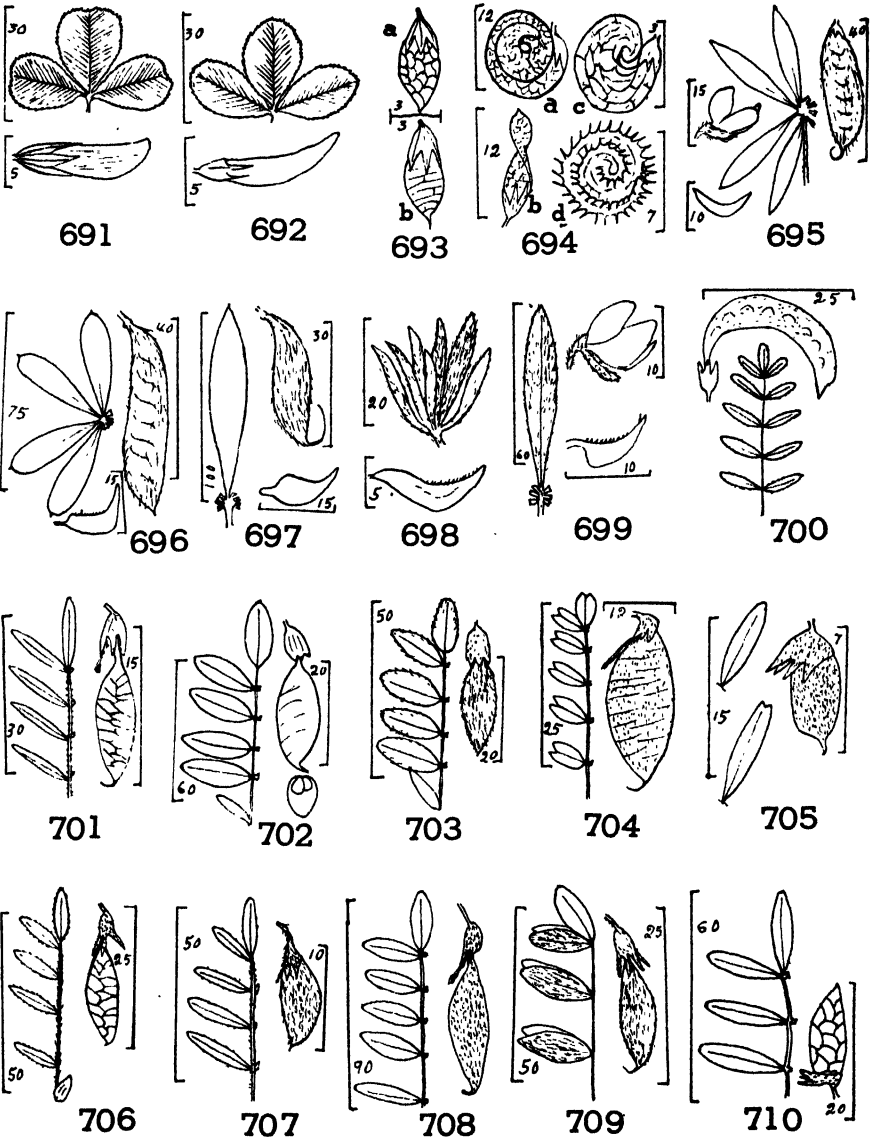
## PLATE XXIX

Scale marked in millimeters

FIG.

691. *Trifolium repens* L. Leaf and flower.  
692. *Trifolium hybridum* L. Leaf and flower.  
693. Pods of *Melilotus*. (a) *M. alba* Desv. (b) *M. officinalis* Lam.  
694. Pods of *Medicago*. (a) *M. sativa* L. (b) *M. falcata* L. (c) *M. lupulina* L. (d) *M. hispida* Gaertn.  
695. *Lupinus arcticus* Wats. Flower, keel, part of leaf, and pod.  
696. *Lupinus nootkatensis* Donn. Part of leaf, keel, and pod.  
697. *Lupinus polyphyllus* Lindl. Leaflet, pod, and keel.  
698. *Lupinus lepidus* Dougl. Leaf and keel.  
699. *Lupinus sericeus* Pursh. Leaflet, flower, and keel.  
700. *Astragalus nutzotenensis* Rousseau. Pod and leaf.  
701. *Astragalus tenellus* Pursh. Part of leaf and pod.  
702. *Astragalus americanus* (Hook.) Jones. Part of leaf and pod.  
703. *Astragalus umbellatus* Bunge. Part of leaf and pod.  
704. *Astragalus polaris* (Seem.) Benth. Part of leaf and pod.  
705. *Astragalus yukonis* Jones. Leaflets and pod.  
706. *Astragalus aboriginorum* Rich. Part of leaf and pod.  
707. *Astragalus eucosmus* Robins. Part of leaf and pod.  
708. *Astragalus macounii* Rydb. Part of leaf and pod.  
709. *Astragalus harringtonii* Cov. & Standl. Part of leaf and pod.  
710. *Astragalus williamsii* Rydb. Part of leaf and pod.

PLATE XXIX



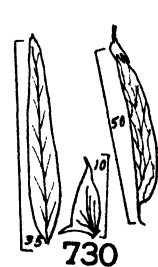
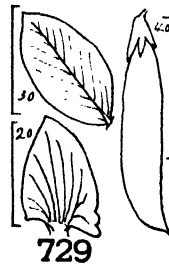
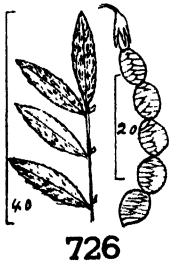
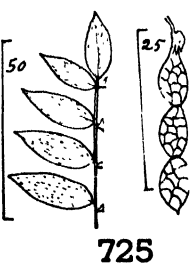
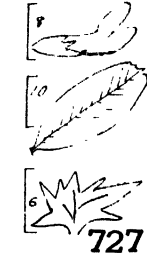
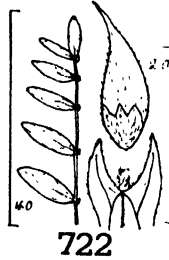
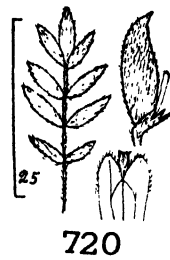
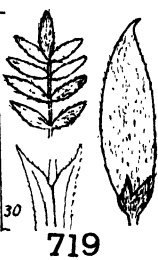
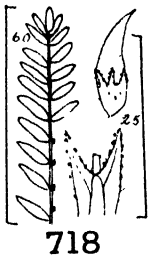
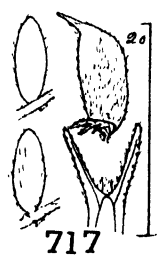
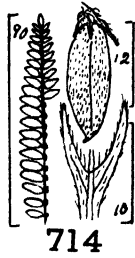
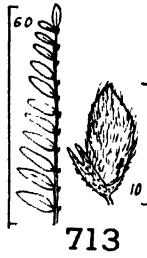
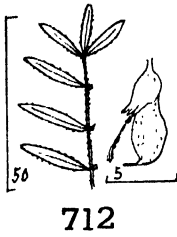
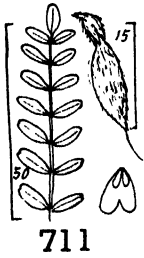
## PLATE XXX

Scale marked in millimeters

FIG.

- 711. *Astragalus alpinus* L. Part of leaf and pod.
- 712. *Astragalus vicifolius* Hult. Part of leaf and pod.
- 713. *Astragalus agrestis* Dougl. Part of leaf and pod.
- 714. *Oxytropis deflexa* (Pall) DC. Part of leaf, pod, and stipule.
- 715. *Oxytropis mertensiana* Turcz. Leaf, pod, and stipule.
- 716. *Oxytropis leucantha* (Pall.) Bunge. Part of leaf, pod, and stipules. These are parts illustrated in the other species of *Oxytropis*.
- 717. *Oxytropus vicida* Nutt.
- 718. *Oxytropis vicidula* (Rydb.) Tidestrom.
- 719. *Oxytropis nigrescens bryophila* (Greene) Hult.
- 720. *Oxytropis scammaniana* Hult.
- 721. *Oxytropus maydeliana* Trautv.
- 722. *Oxytropis gracilis* (A.Nels.) K. Schum.
- 723. *Oxytropis splendens* Dougl.
- 724. *Oxytropis varians* (Rydb.) Hult.
- 725. *Hedysarum alpinum americanum*. (Michx.) Fedtsch. Part of leaf and fruit.
- 726. *Hedysarum mackenzii* Rich. Part of leaf and fruit.
- 727. *Vicia americana* Muhl. Flower, leaflet, and stipule.
- 728. *Vicia gigantea* Hook. Pod, leaflet, and stipule.
- 729. *Lathyrus maritimus* (L.) Bigel. Leaflet, stipule, and pod.
- 730. *Lathyrus palustris pilosus* (Cham.) Hult.

PLATE XXX





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## 3

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